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Supporting Information

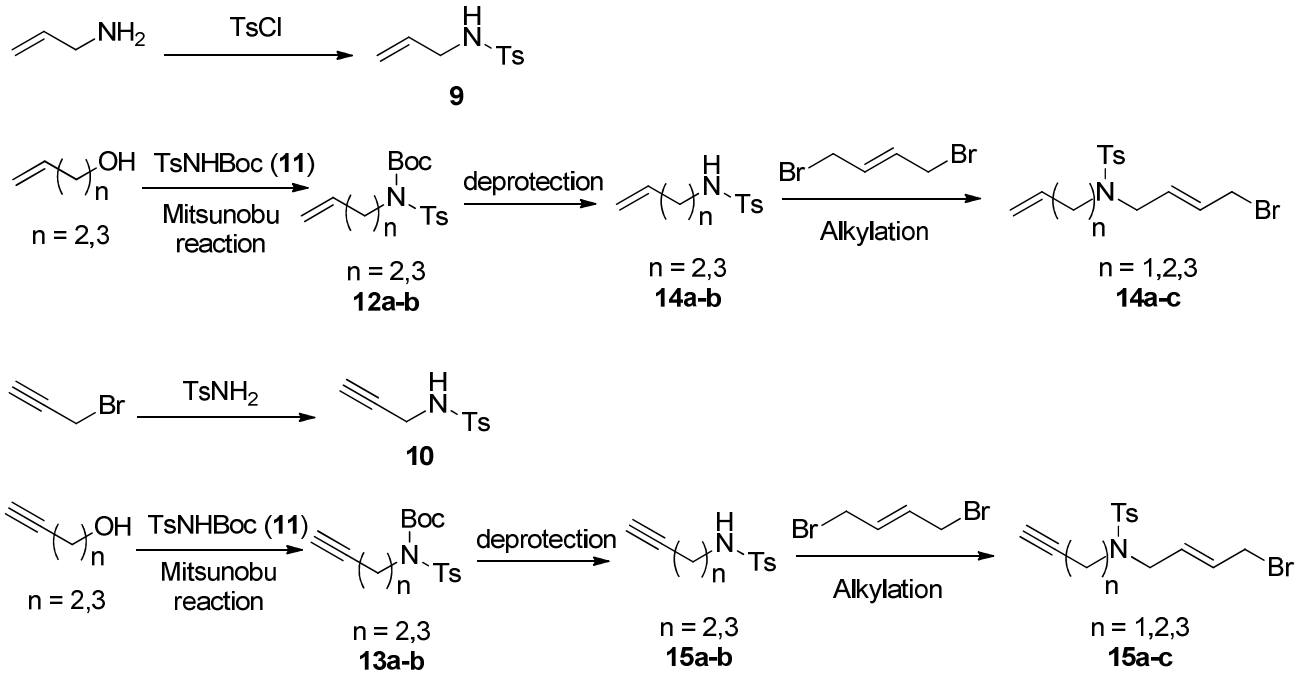
Asymmetric Allylic Alkylation in Combination with Ring Closing Metathesis for the Preparation of Chiral N-Heterocycles

Johannes F. Teichert, Suyan Zhang, Anthoni W. van Zijl, Jan Willem Slaa, Adriaan J. Minnaard and Ben L. Feringa

Stratingh Institute for Chemistry, University of Groningen,

General remarks:

^{1}H NMR and ^{13}C NMR spectra were recorded on a Varian AMX400 (400 and 100 MHz, respectively), a Varian VXR300 (300 and 75 MHz, respectively), or a Varian VXR200 NMR spectrometer (200 MHz and 75 MHz, respectively) with $CDCl_3$ as solvent. Chemical shifts were determined relative to the residual solvent peaks ($CHCl_3$, δ = 7.26 ppm for ^{1}H NMR, δ = 77.0 ppm for ^{13}C NMR). The following abbreviations are used to indicate signal multiplicity: s, singlet; d, doublet; t, triplet; q, quartet; qi, quintet; m, multiplet; br, broad. Enantiomeric excesses were determined by chiral HPLC using a Shimadzu LC-10ADVP HPLC equipped with a Shimadzu SPD-M10AVP diode array detector, in comparison with racemic products or, in some cases, mixtures of both enantiomers. Racemic products were obtained by the same procedure as the enantioselective allylic alkylation only using $CuBr{\cdot}SMe_2$ (10 mol%), PPh_3 (20 mol%) and MeMgBr (1.15 eq.) at -40 °C in CH_2Cl_2. The opposite enantiomer of a product is obtained by using the (S,S_p) enantiomer of **L1**, following the general procedure **D**. Regioselectivities were determined by ^{1}H NMR. Optical rotations were measured on a *Schmidt + Haensch* polarimeter (Polartronic MH8) with a 10 cm cell (*c* given in g/100 mL) at 20 °C. Thin-layer chromatography (TLC) was performed on Merck TLC Silica gel 60 Kieselguhr $F_{254.}$ Flash chromatography was performed on silica gel Merck Type 9385 230-400 mesh. Mass spectra were recorded on a AEI-MS-902 mass spectrometer (EI+) or a LTQ Orbitrap XL (ESI+).



N-allyl-4-methylbenzenesulfonamide (9):

This compound was prepared according to a literature procedure[1] (75% yield, 6.3 g).
^{1}H NMR (300 MHz, $CDCl_3$) δ 7.75 (d, *J* = 8.1 Hz, 2H), 7.29 (d, *J* = 8.0 Hz, 2H), 5.76-5.63 (m, 1H), 5.10 (dd, *J* = 24.9 Hz, 13.7 Hz, 2H), 4.92 (br, 1H), 3.55 (t, *J* = 5.9 Hz, 2H), 2.41 (s, 3H).

^{13}C NMR (75 MHz, $CDCl_3$) δ 145.81, 143.51, 128.66, 127.49, 126.37, 113.35, 43.44, 21.00.
HRMS calcd. For $C_{10}H_{14}NO_2S[M+H^+]$: 212.0745, found 212.0740.

4-Methyl-N-(prop-2-yn-1-yl)benzenesulfonamide (10):

This compound wasa prepared according to a literature procedure[2] (39% yield, 1.64 g).
^{1}H NMR (300 MHz, $CDCl_3$) δ 7.77 (d, *J* = 8.3 Hz, 2H), 7.32 (d, *J* = 8.0 Hz, 2H), 4.53 (br, 1H), 3.83 (dd, *J* = 6.1 Hz, 2.5 Hz, 2H), 2.44 (s, 3H), 2.11 (t, *J* = 2.5 Hz, 1H).
^{13}C NMR (75 MHz, $CDCl_3$) δ 143.83, 139.31, 129.93, 126.68, 98.20, 74.22, 36.39, 21.73.
HRMS calcd. For $C_{10}H_{12}NO_2S[M+H^+]$: 210.0589, found 210.0583.

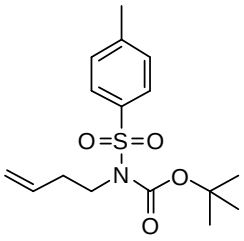
tert-*Butyl tosylcarbamate (11):*

This compound was prepared according to a literature procedure[3] (87% yield, 7.06 g).
^{1}H NMR (400 MHz, $CDCl_3$) δ 7.90 (d, *J* = 7.5 Hz, 2H), 7.34 (d, *J* = 7.6 Hz, 2H), 2.45 (s, 3H), 1.38 (s, 9H).
^{13}C NMR (75 MHz, $CDCl_3$) δ 149.19, 144.97, 136.15, 129.71, 128.46, 84.28, 28.09, 21.88.
HRMS calcd. For $C_{12}H_{18}NO_4S[M+H^+]$: 272.0957, found 272.0951.

General procedure A: Preparation of olefinic and propargylic *N*-Boc protected sulfonamides[4] (12a-b, 13a-b).

N-Boc *p*-toluenesulfonamide (7.37 mmol, 2.0 g, 1.5 eq.) was dissolved in dry THF (3 mL) and triphenylphosphine (14.7 mmol, 3.87 g, 3.0 eq.) was added. The solution was stirred under nitrogen atmosphere and the olefinic or propargylic alcohol (4.9 mmol, 1.0 eq.) was added followed by diethyl azodicarboxylate (12.2 mmol, 2.12 g, 2.5 eq.). The mixture was stirred at room temperature for 3h, concentrated under reduced pressure and the product was purified by flash chromatography (SiO_2).

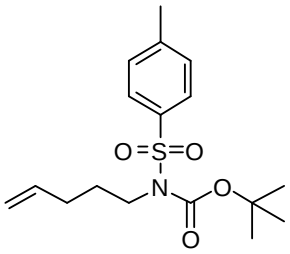
*(*N-tert-*Butoxycarbonyl)(but-3-enyl)tosylamide*[5] *(12a):*



The title compound was prepared from 3-buten-1-ol (5.5 mmol, 0.40 g) following general procedure **A**. Purification by column chromatography (SiO_2, 1:8 EtOAc/heptane, R_f (1:5 EtOAc/heptane) = 0.54) afforded product as a yellow oil (86% yield, 1.54 g).
^{1}H NMR (200 MHz, $CDCl_3$) δ 7.79 (d, *J* = 8.4 Hz, 2H), 7.30 (d, *J* = 8.1 Hz, 2H), 5.91 – 5.71 (m, 1H), 5.20 – 5.02 (m, 2H), 3.92 – 3.85 (m, 2H), 2.58 – 2.46 (m, 2H), 2.44 (s, 3H), 1.34 (s, 9H).
^{13}C NMR (75 MHz, $CDCl_3$) δ 151.14, 144.26, 137.74, 134.62, 129.42, 128.08, 117.64, 84.34, 46.60, 34.80, 28.09, 21.81.

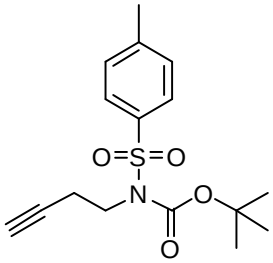
HRMS calcd. For $C_{16}H_{23}NO_4SNa$ [M+Na^+]: 348.1245, found 348.1240.

*(*N-tert-*Butoxycarbonyl)(but-3-ynyl)tosylamide (12b):*



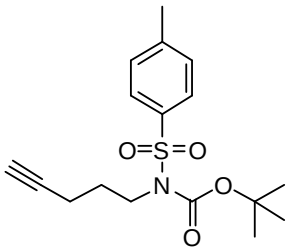
The title compound was prepared from 4-penten-1-ol (0.81 mmol, 70 mg) following general procedure **A**. Purification by column chromatography (SiO_2, 1:8 EtOAc/pentane, R_f (1:6 EtOAc/pentane) = 0.44) afforded product as a colourless oil (97% yield, 266 mg).
^{1}H NMR (200 MHz, $CDCl_3$) δ 7.74 (d, *J* = 8.4 Hz, 2H), 7.27 (d, *J* = 8.0 Hz, 2H), 5.81 (ddt, *J* = 16.7 Hz, 10.2 Hz, 6.5 Hz, 1H), 5.11 – 4.91 (m, 2H), 3.85 – 3.73 (m, 2H), 2.40 (s, 3H), 2.16 – 2.05 (m, 2H), 1.91 – 1.80 (m, 2H), 1.30 (s, 9H).
^{13}C NMR (50 MHz, $CDCl_3$) δ 150.91, 144.02, 137.47, 137.41, 129.19, 127.72, 115.17, 84.04, 46.71, 30.82, 29.18, 27.83, 21.54.
HRMS calcd. For $C_{17}H_{26}NO_4S$[M+H^+]: 340.1583, found 340.1577.

*(*N-tert-*Butoxycarbonyl)(but-3-ynyl)tosylamide*[6] *(13a):*



The title compound was prepared from 3-butyn-1-ol (1.4 mmol, 95 mg) following general procedure **A**. Purification by column chromatography (SiO_2, 5:1 heptane/EtOAc, R_f = 0.24) afforded product as an opaque oil (89% yield, 391 mg).
^{1}H NMR (300 MHz, $CDCl_3$) δ 7.80 (d, *J* = 8.2 Hz, 2H), 7.31 (d, *J* = 8.1 Hz, 2H), 4.03 – 3.98 (m, 2H), 2.69 – 2.63 (m, 2H), 2.44 (s, 3H), 2.03 – 2.01 (m, 1H), 1.35 (s, 9H).
^{13}C NMR (75 MHz, $CDCl_3$) δ 150.93, 144.46, 137.45, 129.47, 128.11, 84.74, 80.65, 70.60, 45.42, 28.06, 21.82, 20.21.
HRMS calcd. For $C_{16}H_{21}NO_4SNa$ [M+Na^+]: 346.1089, found 346.1084.

*(*N-tert-*Butoxycarbonyl)(pent-4-ynyl)tosylamide (13b):*

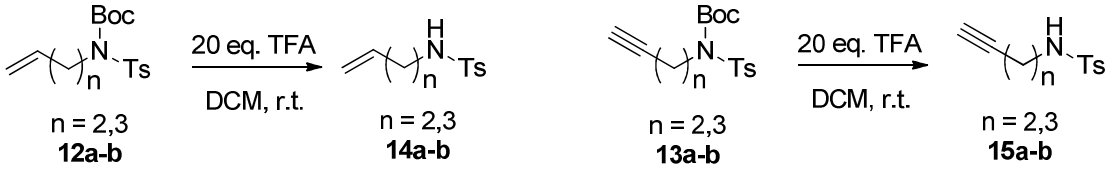


The title compound was prepared from 4-pentyn-1-ol (0.64 mmol, 54 mg) following general procedure **A**. Purification by column chromatography (SiO_2, 5:1 heptane/EtOAc, R_f = 0.27) afforded product as an opaque oil (76% yield, 165 mg).
^{1}H NMR (400 MHz, $CDCl_3$) δ 7.76 (d, *J* = 8.2 Hz, 2H), 7.28 (d, *J* = 8.0 Hz, 2H), 3.90 (t, *J* = 8.0 Hz, 2H), 2.41 (s, 3H), 2.26 (td, *J* = 7.1 Hz, 2.4 Hz, 2H), 1.97 (m, 3H), 1.32 (s, 9H).
^{13}C NMR (75 MHz, $CDCl_3$) δ 151.07, 144.36, 137.53, 129.46, 128.00, 84.42, 83.24, 69.23, 46.48, 29.10, 28.05, 21.75, 16.20.
HRMS calcd. For $C_{17}H_{24}NO_4S$[M+H^+]: 338.1426, found 338.4430.

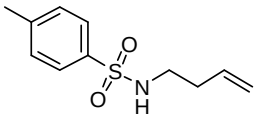
General procedure B: Preparation of olefinic and propargylic tosylamides[7] (14a-b, 15a-b).

To a solution of the N-Boc olefinic or propargylic tosylamide (0.62 mmol, 1.0 eq.) in CH_2Cl_2 (10 mL) was added trifluoroacetic acid (12.4 mmol, 1.41 g, 20 eq.) at 0 °C, and the mixture was stirred at rt for 3 h. The mixture was diluted with EtOAc, and the

organic layer was washed with saturated $NaHCO_3$ solution and saturated NaCl solution, dried and concentrated to afford the products as colourless oils.

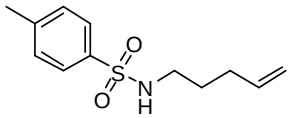


N-*3-Buten-1-yl-4-methyl-benzenesulfonamide*[8] *(14a):*



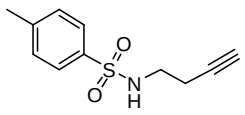
The title compound was prepared from **12a** (4.74 mmol, 1.54 g) following general procedure **B** (70% yield, 744 mg).
^{1}H NMR (300 MHz, $CDCl_3$) δ 7.74 (d, *J* = 8.2 Hz, 2H), 7.30 (d, *J* = 8.0 Hz, 2H), 5.69 – 5.55 (m, 1H), 5.07 – 4.98 (m, 2H), 4.66 (t, *J* = 5.7 Hz, 1H), 3.00 (q, *J* = 6.6 Hz, 2H), 2.42 (s, 3H), 2.19 (q, *J* = 6.7 Hz, 2H).
^{13}C NMR (75 MHz, $CDCl_3$) δ 143.63, 137.21, 134.39, 129.92, 127.34, 118.31, 42.31, 33.83, 21.73.
HRMS calcd. For $C_{11}H_{16}NO_2S[M+H^+]$: 226.0902, found 226.0896.

N-*4-penten-1-yl-4-methyl-benzenesulfonamide*[8] *(14b):*



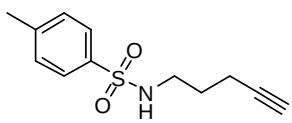
The title compound was prepared from **12b** (0.77 mmol, 260 mg) following general procedure **B** (86% yield, 157 mg).
^{1}H NMR (400 MHz, $CDCl_3$) δ 7.75 (d, *J* = 8.3 Hz, 2H), 7.29 (d, *J* = 8.1 Hz, 2H), 5.68 (ddt, *J* = 16.9 Hz, 10.2 Hz, 6.7 Hz, 1H), 5.01 (t, *J* = 6.1 Hz, 1H), 4.97 – 4.89 (m, 2H), 2.91 (dd, *J* = 13.5 Hz, 6.8 Hz, 2H), 2.41 (s, 3H), 2.02 (q, *J* = 7.2 Hz, 2H), 1.54 (qi, *J* = 7.2 Hz, 2H).
^{13}C NMR (100 MHz, $CDCl_3$) δ 143.29, 137.25, 136.95, 129.66, 127.06, 115.44, 42.58, 30.60, 28.63, 21.48.
HRMS calcd. For $C_{12}H_{17}NO_2SNa$ $[M+Na^+]$: 262.0878, found 262.0872.

N-*3-butyn-1-yl-4-methyl-benzenesulfonamide*[9] *(15a):*



The title compound was prepared from **13a** (0.62 mmol, 200 mg) following general procedure **B** (74% yield, 103 mg).
^{1}H NMR (300 MHz, $CDCl_3$) δ 7.75 (d, *J* = 8.3 Hz, 2H), 7.29 (d, *J* = 8.1 Hz, 2H), 5.13 (t, *J* = 6.2 Hz, 1H), 3.08 (q, *J* = 6.6 Hz, 2H), 2.41 (s, 3H), 2.32 (td, *J* = 6.7 Hz, 2.6 Hz, 2H), 1.98 (t, *J* = 2.6 Hz, 1H).
^{13}C NMR (75 MHz, $CDCl_3$) δ 143.81, 137.13, 130.00, 127.29, 80.61, 71.02, 41.89, 21.74, 20.00.
HRMS calcd. For $C_{11}H_{14}NO_2S[M+H^+]$: 224.0745, found 224.0740.

N-*4-pentyn-1-yl-4-methyl-benzenesulfonamide*[10] *(15b):*

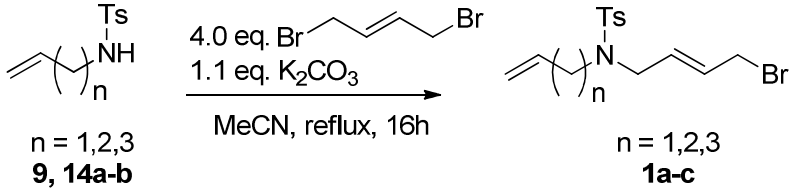


The title compound was prepared from **13b** (0.50 mmol, 170 mg) following general procedure **B** (83% yield, 99 mg).
^{1}H NMR (300 MHz, $CDCl_3$) δ 7.75 (d, *J* = 8.2 Hz, 2H), 7.31 (d, *J* = 8.0 Hz, 2H), 4.54 (br, 1H), 3.08 (q, *J* = 6.6 Hz, 2H), 2.43 (s, 3H), 2.22 (td, *J* = 6.8 Hz, 2.5 Hz, 2H), 1.95 (s, 1H), 1.69 (qi, *J* = 6.8 Hz, 2H).

^{13}C NMR (100 MHz, $CDCl_3$) δ 143.42, 136.86, 129.71, 127.07, 82.89, 69.39, 42.11, 28.12, 21.50, 15.69.
HRMS calcd. For $C_{12}H_{16}NO_2S[M+H^+]$: 238.0902, found 238.0896.

General procedure C: Preparation of allylic bromide substrates (1a-c, 5a-c).

To a suspension of olefinic or propargylic tosylamide (19.2 mmol, 1.0 eq.) and K_2CO_3 (28.8 mmol, 3.98 g, 1.1 eq.) in 20 mL MeCN was added 1,4-dibromobut-2-ene (77.0 mmol, 16.5 g, 4.0 eq.) and the mixture was heated to reflux for 24 h. The mixture was then concentrated under reduced pressure and water (10 mL) and Et_2O (10 mL) were added. The organic layer was separated and the aqueous layer was extracted with Et_2O (2 x 5 mL). The combined organic layers were dried, filtered and concentrated under reduced pressure. Purification by column chromatography (SiO_2) yielded desired products.



(E)-N-*(4-bromo-2-buten-1-yl)-4-methyl*-N-*2-propen-1-yl-benzenesulfonamide*[11] *(1a):*

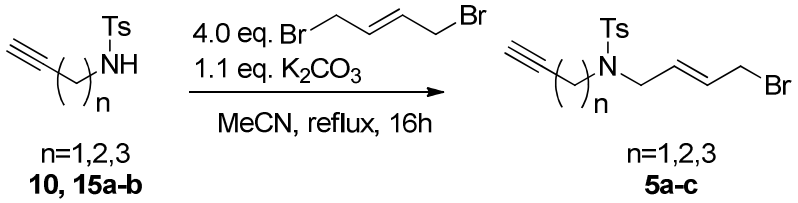
The title compound was prepared from **9** (19.2 mmol, 4.06 g) following general procedure **C**. Purification by column chromatography (SiO_2, 1:5 EtOAc/Pentane, R_f = 0.38) afforded **1a** (70% yield, 4.63 g) as an opaque oil.
^{1}H NMR (200 MHz, $CDCl_3$) δ 7.68 (d, *J* = 8.3 Hz, 2H), 7.29 (d, *J* = 8.0 Hz, 2H), 5.85 – 5.68 (m, 1H), 5.66 – 5.43 (m, 2H), 5.20 – 5.08 (m, 2H), 3.85 (d, *J* = 7.3 Hz, 2H), 3.78 (d, *J* = 6.2 Hz, 4H), 2.41 (s, 3H).
^{13}C NMR (75 MHz, $CDCl_3$) δ 143.63, 137.36, 132.73, 130.71, 129.99, 129.84, 127.38, 119.57, 49.94, 47.94, 31.67, 21.75.
HRMS calcd. For $C_{14}H_{19}BrNO_2S[M+H^+]$: 344.0320, found 344.0314.

(E)-N-*(4-bromo-2-buten-1-yl)-4-methyl*-N-*3-buten-1-yl-benzenesulfonamide (1b):*

The title compound was prepared from **14a** (2.87 mmol, 734 mg) following general procedure **C**. Purification by column chromatography (SiO_2, 1:5 EtOAc/Pentane, R_f = 0.23) afforded **1b** (74% yield, 762 mg) as an opaque oil.
^{1}H NMR (300 MHz, $CDCl_3$) δ 7.68 (d, *J* = 8.2 Hz, 2H), 7.30 (d, *J* = 8.1 Hz, 2H), 5.87 – 5.76 (m, 1H), 5.74 – 5.54 (m, 2H), 5.11 – 4.98 (m, 2H), 3.87 (d, *J* = 7.4 Hz, 2H), 3.81 (d, *J* = 6.3 Hz, 2H), 3.17 (t, *J* = 6.5 Hz, 2H), 2.42 (s, 3H), 2.27 (q, *J* = 6.4 Hz, 2H).
^{13}C NMR (75 MHz, $CDCl_3$) δ 143.56, 137.15, 134.77, 130.42, 130.37, 129.94, 127.39, 117.41, 49.37, 47.32, 33.22, 31.51, 21.73.
HRMS calcd. For $C_{15}H_{21}BrNO_2S[M+H^+]$: 358.0476, found 358.0471.

(E)-N-*(4-bromo-2-buten-1-yl)-4-methyl*-N-*4-penten-1-yl-benzenesulfonamide (1c):*

The title compound was prepared from **14b** (0.648 mmol, 155 mg) following general procedure **C**. Purification by column chromatography (SiO_2, 1:7 Et_2O/Pentane, R_f (1:6 Et_2O/Pentane) = 0.23) afforded **1c** (47% yield, 116 mg) as an opaque oil.
^{1}H NMR (400 MHz, $CDCl_3$) δ 7.67 (d, *J* = 8.3 Hz, 2H), 7.29 (d, *J* = 8.1 Hz, 2H), 5.87 – 5.69 (m, 2H), 5.65 – 5.55 (m, 1H), 5.04 – 4.92 (m, 2H), 3.86 (d, *J* = 7.4 Hz, 2H), 3.78 (d, *J* = 6.5 Hz, 2H), 3.09 (t, *J* = 8.0 Hz, 2H), 2.42 (s, 3H), 2.02 (dd, *J* = 14.2 Hz, 7.2 Hz, 2H), 1.64 – 1.57 (m, 2H).
^{13}C NMR (100 MHz, $CDCl_3$) δ 143.28, 137.42, 136.83, 130.24, 130.09, 129.70, 127.13, 115.27, 49.10, 47.25, 31.33, 30.66, 27.49, 21.49.
HRMS calcd. For $C_{16}H_{23}BrNO_2S$[M+H$^+$]: 372.0633, found 372.0627.



n=1,2,3
10, 15a-b

n=1,2,3
5a-c

(E)-N-*(4-bromo-2-buten-1-yl)-4-methyl*-N-*2-propyn-1-yl-benzenesulfonamide*[12] *(5a):*

The title compound was prepared from **10** (1.20 mmol, 250 mg) following general procedure **C**. Purification by column chromatography (SiO_2, 1:15 EtOAc/Heptane, R_f (1:9 EtOAc/Heptane) = 0.25) afforded **5a** (73% yield, 299 mg) as a colourless oil.
^{1}H NMR (400 MHz, $CDCl_3$) δ 7.73 (d, *J* = 8.3 Hz, 2H), 7.30 (d, *J* = 8.4 Hz, 2H), 5.98-5.90 (m, 1H), 5.75 – 5.64 (m, 1H), 4.09 (d, *J* = 2.4 Hz, 2H), 3.92 (d, *J* = 7.5 Hz, 2H), 3.85 (d, *J* = 6.5 Hz, 2H), 2.43 (s, 3H), 2.03 (t, *J* = 2.5 Hz, 1H).
^{13}C NMR (100 MHz, $CDCl_3$) δ 143.73, 135.75, 131.46, 129.55, 128.71, 127.72, 76.31, 74.01, 47.39, 36.03, 31.15, 21.57.
HRMS calcd. For $C_{14}H_{16}BrNO_2SNa$[M+Na$^+$]: 363.9983, found 363.9977.

(E)-N-*(4-bromo-2-buten-1-yl)-4-methyl*-N-*3-butyn-1-yl-benzenesulfonamide (5b):*

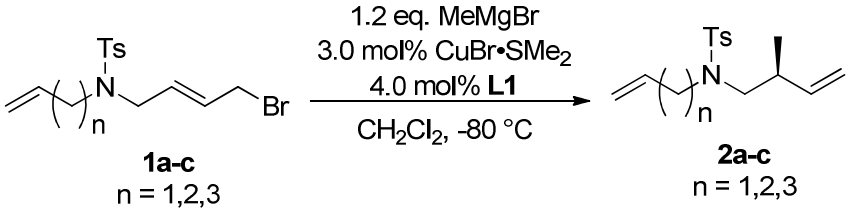
The title compound was prepared from **15a** (0.34 mmol, 76 mg) following general procedure **C**. Purification by column chromatography (SiO_2, 1:7 EtOAc/Heptane, R_f (1:5 EtOAc/Heptane) = 0.22) afforded **5b** (71% yield, 85 mg) as an opaque oil.
^{1}H NMR (300 MHz, $CDCl_3$) δ 7.69 (d, *J* = 8.1 Hz, 2H), 7.31 (d, *J* = 8.1 Hz, 2H), 5.84 (dt, *J* = 14.8 Hz, 7.4 Hz, 1H), 5.69 – 5.55 (m, 1H), 3.93 – 3.81 (m, 4H), 3.28 (t, *J* = 7.4 Hz, 2H), 2.46 (m, 2H), 2.43 (s, 3H), 1.97 (t, *J* = 2.4 Hz, 1H).
^{13}C NMR (75 MHz, $CDCl_3$) δ 143.79, 136.93, 130.76, 130.11, 130.03, 127.40, 81.13, 70.51, 49.92, 46.53, 31.32, 21.74, 19.66.
HRMS calcd. For $C_{15}H_{19}BrNO_2S$[M+H$^+$]: 356.0320, found 356.0314.

(E)-N-*(4-bromo-2-buten-1-yl)-4-methyl*-N-*4-pentyn-1-yl-benzenesulfonamide (5c):*

The title compound was prepared from **15b** (0.12 mmol, 28 mg) following general procedure **C**. Purification by column chromatography (SiO_2, 1:8 EtOAc/Heptane, R_f (1:5 EtOAc/Heptane) = 0.29) afforded **5c** (72% yield, 78 mg) as an opaque oil.
^{1}H NMR (400 MHz, $CDCl_3$) δ 7.68 (d, *J* = 8.3 Hz, 2H), 7.30 (d, *J* = 8.0 Hz, 2H), 5.83 (dt, *J* = 15.0 Hz, 7.5 Hz, 1H), 5.66 – 5.55 (m, 1H), 3.87 (d, *J* = 7.5 Hz, 2H), 3.80 (d, *J* = 6.5 Hz, 2H), 3.20 (t, *J* = 7.2 Hz, 2H), 2.42 (s, 3H), 2.20 (dt, *J* = 7.0 Hz, 2.6 Hz, 2H), 1.95 (t, *J* = 2.6 Hz, 1H), 1.75 (qi, *J* = 7.2 Hz, 2H).
^{13}C NMR (100 MHz, $CDCl_3$) δ 143.41, 136.60, 130.44, 129.96, 129.75, 127.18, 83.13, 69.11, 49.49, 46.65, 31.25, 27.38, 21.51, 15.73.
HRMS calcd. For $C_{16}H_{20}BrNO_2SNa[M+Na^+]$: 392.0296, found 392.0290.

General procedure D: Enantioselective Cu-catalyzed allylic alkylation with methylmagnesium bromide (2a-c, 6a-c).

In a dry Schlenk tube equipped with septum and stirring bar, $CuBr{\cdot}SMe_2$ (15 μmol, 3.1 mg, 1.0 mol%) and **L1** (18 μmol, 12.4 mg, 1.2 mol%) were dissolved in CH_2Cl_2 (2.0 mL) and stirred under nitrogen atmosphere at room temperature for 10 min. The mixture was cooled to -80 °C and a solution of methylmagnesium bromide (1.73 mmol, 3M solution in Et_2O, 1.15 eq.) in 1.0 mL CH_2Cl_2 was added dropwise over 20 min via syringe pump. Subsequently, a solution of allylic bromide (1.5 mmol) in 1.0 mL CH_2Cl_2 was added dropwise over 30 min via syringe pump. Once the addition was complete, the resulting mixture was stirred at -80 °C for 16h. The reaction was quenched by addition of MeOH (2.0 mL) and was allowed to warm up to rt. Aqueous NH_4Cl solution (1M, 10 mL) was added and the organic phase separated. The aqueous phase was extracted with Et_2O (2 x 10 mL). The combined organic phases were dried over $MgSO_4$ and concentrated under reduced pressure to yield the crude product which was purified by flash chromatography SiO_2.



(S)-N-*Allyl-4-methyl*-N-*(2-methylbut-3-en-1-yl)benzenesulfonamide (2a):*

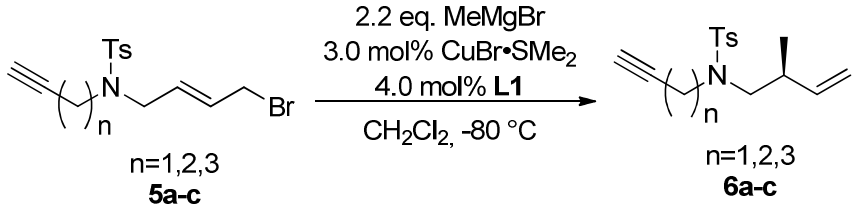
The title compound was prepared from **1a** (1.50 mmol, 516 mg) following general procedure **D**. Purification by column chromatography (SiO_2, EtOAc/Pentane 1:9, R_f = 0.44) afforded **2a** (74% yield, 277 mg, ratio **2a:3a** = 95:5, 99% *ee*, $[\alpha]_D$ = -1.1 (*c* 17.4, $CHCl_3$)) as a yellow oil.
Enantiomeric excess determined by chiral HPLC analysis, Chiralpak AD (99% *n*-heptane/1% *i*-PrOH), 40 °C, retention times (min) 16.1 (major) and 17.4 (minor).
^{1}H NMR (300 MHz, $CDCl_3$) δ 7.65 (d, *J* = 8.1 Hz, 2H), 7.25 (d, *J* = 8.0 Hz, 2H), 5.75 – 5.41 (m, 2H), 5.13– 4.92 (m, 4H), 3.76 (d, *J* = 6.4 Hz, 2H), 3.10 – 2.86 (m, 2H), 2.51 – 2.42 (m, 1H), 2.36 (s, 3H), 0.96 (d, *J* = 6.7 Hz, 3H).
^{13}C NMR (75 MHz, $CDCl_3$) δ 143.38, 141.27, 137.31, 133.25, 129.85, 127.38, 119.11, 115.01, 52.92, 51.32, 36.71, 21.67, 17.62.
HRMS calcd. For $C_{15}H_{22}NO_2S[M+H^+]$: 280.1371, found 280.1366.

(S)-N-*(but-3-en-1-yl)-4-methyl*-N-*(2-methylbut-3-en-1-yl)benzenesulfonamide (2b):*

The title compound was prepared from **1b** (0.017 mmol, 3.4 mg) following general procedure **D**. Purification by column chromatography (SiO_2, EtOAc/Pentane 1:7, R_f (EtOAc/Pentane 1:5) = 0.64) afforded **2b** (84% yield, 69 mg, ratio **2b:3b** = 98:2, 90% *ee*, $[\alpha]_D$ = +1.2 (*c* 0.5, $CHCl_3$)) as a yellow oil.
Enantiomeric excess determined by chiral HPLC analysis, Chiralcel OJ (99% *n*-heptane/1% *i*-PrOH), 40 °C, retention times (min) 9.3 (major) and 11.6 (minor).
^{1}H NMR (300 MHz, $CDCl_3$) δ 7.68 (d, *J* = 8.2 Hz, 2H), 7.28 (d, *J* = 8.1 Hz, 2H), 5.76 – 5.59 (m, 2H), 5.07 – 4.95 (m, 4H), 3.15 (m, 2H), 3.09 – 2.93 (m, 2H), 2.54 – 2.44 (m, 1H), 2.41 (s, 3H), 2.32 – 2.17 (m, 2H), 1.02 (d, *J* = 6.7 Hz, 3H).
^{13}C NMR (75 MHz, $CDCl_3$) δ 143.30, 141.21, 137.20, 134.94, 129.79, 127.44, 117.14, 115.13, 54.26, 48.47, 37.06, 33.16, 21.69, 17.70.
HRMS calcd. For $C_{16}H_{24}NO_2S[M+H^+]$: 294.1528, found 294.1522.

(S)-4-methyl-N-*(2-methylbut-3-en-1-yl)*-N-*(pent-4-en-1-yl)benzenesulfonamide (2c):*

The title compound was prepared from **1c** (0.11 mmol, 42 mg) following general procedure **D**. Purification by column chromatography (SiO_2, Et_2O/Pentane 1:8, R_f = 0.39) afforded **2c** (72% yield, 25 mg, ratio **2c:3c** = 92:8, 98% *ee*, $[\alpha]_D$ = +0.7 (*c* 0.8, $CHCl_3$)) as a yellow oil. Enantiomeric excess determined by chiral HPLC analysis, Chiralpak OD-H (99.5% *n*-heptane/0.05% *i*-PrOH), 40 °C, retention times (min) 45.4 (minor) and 47.9 (major).
^{1}H NMR (400 MHz, $CDCl_3$) δ 7.68 (d, *J* = 8.3 Hz, 2H), 7.29 (d, *J* = 8.0 Hz, 2H), 5.80 – 5.63 (m, 2H), 5.06 – 4.94 (m, 4H), 3.11 – 3.05 (m, 2H), 3.05 – 2.94 (m, 2H), 2.53 – 2.43 (m, 1H), 2.42 (s, 3H), 2.00 (dd, *J* = 14.2 Hz, 7.4 Hz, 2H), 1.67 – 1.57 (m, 2H), 1.02 (d, *J* = 6.7 Hz, 3H).
^{13}C NMR (75 MHz, $CDCl_3$) δ 143.22, 141.27, 137.67, 137.14, 129.76, 127.45, 115.47, 115.08, 54.35, 48.71, 37.17, 31.13, 27.75, 21.69, 17.72.
HRMS calcd. For $C_{17}H_{25}NO_2SNa[M+Na^+]$: 330.1504, found 330.1498.



(S)-4-methyl-N-(2-methylbut-3-en-1-yl)-N-(prop-2-yn-1-yl)benzenesulfonamide (6a):

The title compound was prepared from **5a** (22 mmol, 75 mg) following general procedure **D**. Purification by column chromatography (SiO_2, EtOAc/Pet-Ether 40-60 1:9, R_f = 0.59) afforded **6a** (77% yield, 47 mg, 99% *ee*, $[\alpha]_D$ = -4.9 (*c* 1.4, $CHCl_3$)) as a colourless oil.

Enantiomeric excess determined by chiral HPLC analysis, Chiralpak AD (99% *n*-heptane/1% *i*-PrOH), 40 °C, retention times (min) 17.6 (major) and 19.1 (minor).
^{1}H NMR (300 MHz, $CDCl_3$) δ 7.71 (d, *J* = 8.3 Hz, 2H), 7.28 (d, *J* = 8.1 Hz, 2H), 5.72 (ddd, *J* = 17.5 Hz, 10.3 Hz, 7.5 Hz, 1H), 5.11 – 5.01 (m, 2H), 4.20 – 4.06 (m, 2H), 3.14 – 3.02 (m, 2H), 2.57 – 2.44 (m, 1H), 2.41 (s, 3H), 1.99 (t, *J* = 2.5 Hz, 1H), 1.04 (d, *J* = 6.7 Hz, 3H).
^{13}C NMR (75 MHz, $CDCl_3$) δ 143.63, 141.04, 136.21, 129.62, 127.95, 115.28, 76.68, 73.99, 51.74, 36.92, 36.35, 21.73, 17.71.
HRMS calcd. For $C_{15}H_{20}NO_2S[M+H^+]$: 278.1215, found 278.1209.

(S)-N-(but-3-yn-1-yl)-4-methyl-N-(2-methylbut-3-en-1-yl)benzenesulfonamide (6b):

The title compound was prepared from **5b** (84 μmol, 30 mg) following general procedure **D**. Purification by column chromatography (SiO_2, 1:7 Et_2O/Pet-Ether 40-60, R_f (1:5 Et_2O/Pet-Ether 40-60) = 0.43) afforded **6b** (53% yield, 13 mg, 99% *ee*, $[\alpha]_D$ = -1.4 (*c* 1.0, $CHCl_3$)) as an opaque oil.
Enantiomeric excess determined by chiral HPLC analysis, Chiralpak AD (95% *n*-heptane/5% *i*-PrOH), 40 °C, retention times (min) 8.1 (major) and 9.6 (minor).
^{1}H NMR (400 MHz, $CDCl_3$) δ 7.69 (d, *J* = 7.9 Hz, 2H), 7.29 (d, *J* = 8.0 Hz, 2H), 5.74 – 5.59 (m, 1H), 5.08 – 4.93 (m, 2H), 3.33 – 3.20 (m, 2H), 3.12 – 2.99 (m, 2H), 2.54 – 2.47 (m, 1H), 2.47 – 2.43 (m, 2H), 2.41 (s, 3H), 1.99 – 1.92 (m, 1H), 1.01 (d, *J* = 6.7 Hz, 3H).
^{13}C NMR (100 MHz, $CDCl_3$) δ 143.58, 141.03, 136.78, 129.91, 127.45, 115.39, 81.22, 70.44, 54.82, 47.94, 37.17, 21.73, 19.47, 17.73.
HRMS calcd. For $C_{16}H_{22}NO_2S[M+H^+]$: 292.1371, found 292.1366.

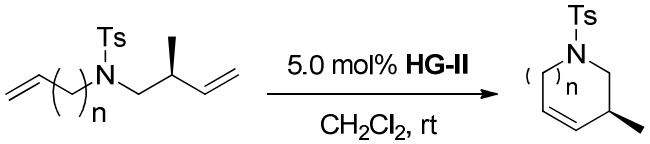
(S)-4-methyl-N-(2-methylbut-3-en-1-yl)-N-(pent-4-yn-1-yl)benzenesulfonamide (6c):

The title compound was prepared from **5c** (0.26 mmol, 80 mg) following general procedure **D**. Purification by column chromatography (SiO_2, 1:7 EtOAc/Heptane, R_f (1:6 EtOAc/Heptane) = 0.45) afforded **6c** (82% yield, 66 mg, 99% *ee*, $[\alpha]_D$ = -3.2 (*c* 1.1, $CHCl_3$)) as an opaque oil.
Enantiomeric excess determined by chiral HPLC analysis, Chiralcel OJ (97% *n*-heptane/3% *i*-PrOH), 40 °C, retention times (min) 11.7 (major) and 13.7 (minor).
^{1}H NMR (200 MHz, $CDCl_3$) δ 7.69 (d, *J* = 8.3 Hz, 2H), 7.29 (d, *J* = 8.0 Hz, 2H), 5.69 (ddd, *J* = 17.5 Hz, 10.3 Hz, 7.4 Hz, 1H), 5.10 – 4.95 (m, 2H), 3.25 – 3.13 (m, 2H), 3.12 – 2.91 (m, 2H), 2.61 – 2.45 (m, 1H), 2.42 (s, 3H), 2.18 (td, *J* = 6.9 Hz, 2.6 Hz, 2H), 1.96 (t, *J* = 2.6 Hz, 1H), 1.85 – 1.67 (m, 2H), 1.02 (d, *J* = 6.7 Hz, 3H).
^{13}C NMR (50 MHz, $CDCl_3$) δ 143.13, 140.97, 136.60, 129.59, 127.25, 115.00, 69.03, 54.52, 48.02, 44.41, 36.91, 27.35, 21.47, 17.55, 15.92.
HRMS calcd. For $C_{17}H_{23}BrNO_2SNa[M+Na^+]$: 328.1347, found 328.1342.

General procedure E: Ru-catalyzed olefin ring-closing metathesis (4a-c).

Substrate (**2a-c**) was dissolved in degassed CH_2Cl_2 (5 mL) and Hoveyda-Grubbs 2nd generation catalyst (5.0 mol%) was added to the solution under a N_2 atmosphere. The mixture was stirred at rt until full conversion (3h) was achieved, as judged by TLC.

The mixture was concentrated under reduced pressure and purified by column chromatography to yield the desired product **4a-c** as colourless oils.



2a-c
n = 1,2,3

4a-c
n = 1,2,3

(S)-3-Methyl-1-tosyl-1,2,3,6-tetrahydropyridine (4a):

The title compound was prepared from **2a** (0.80 mmol, 223 mg) following general procedure **E**. Purification by column chromatography (SiO_2, 1:9 EtOAc/Heptane, R_f (1:5 EtOAc/Heptane) = 0.45) afforded **4a** (54% yield, 80 mg, 99% *ee*, $[\alpha]_D$ = -0.4 (*c* 5.2, $CHCl_3$)) as a colourless oil.

Enantiomeric excess determined by chiral HPLC analysis, Chiralpak AS-H (95% *n*-heptane/5% *i*-PrOH), 40 °C, retention times (min) 17.1 (minor) and 17.8 (major).

^{1}H NMR (300 MHz, $CDCl_3$) δ 7.66 (d, *J* = 7.1 Hz, 2H), 7.31 (d, *J* = 7.8 Hz, 2H), 5.63 – 5.54 (m, 2H), 3.68 (d, *J* = 16.4 Hz, 1H), 3.45 – 3.33 (m, 1H), 2.55 – 2.45 (m, 2H), 2.42 (s, 3H), 0.99 (d, *J* = 6.4 Hz, 3H).

^{13}C NMR (75 MHz, $CDCl_3$) δ 143.68, 133.56, 131.63, 129.84, 127.85, 121.84, 49.60, 44.94, 30.49, 21.72, 18.49.

HRMS calcd. For $C_{13}H_{18}NO_2S[M+H^+]$: 252.1058, found 252.1053.

(S)-3-methyl-1-tosyl-2,3,6,7-tetrahydro-1H-azepine (4b):

The title compound was prepared from **2b** (0.14 mmol, 34 mg) following general procedure **E**. Purification by column chromatography (SiO_2, 1:9 EtOAc/Heptane, R_f = 0.34) afforded **4b** (61% yield, 19 mg, 90% *ee*, $[\alpha]_D$ = -1.8 (*c* 1.0, $CHCl_3$)) as a colourless oil.

Enantiomeric excess determined by chiral HPLC analysis, Chiralpak OJ-H (99% *n*-heptane/1% *i*-PrOH), 40 °C, retention times (min) 40.9 (minor) and 42.2 (major).

^{1}H NMR (400 MHz, $CDCl_3$) δ 7.69 – 7.63 (m, 2H), 7.30 – 7.28 (m, 2H), 5.69 – 5.62 (m, 1H), 5.55 – 5.49 (m, 1H), 3.55 – 3.45 (m, 2H), 2.97 (ddd, *J* = 13.1 Hz, 7.4 Hz, 4.0 Hz, 1H), 2.76 (dd, *J* = 13.0 Hz, 9.1 Hz, 1H), 2.57 (br, 1H), 2.42 (s, 3H), 2.35 – 2.25 (m, 2H), 1.05 (d, *J* = 7.2 Hz, 3H).

^{13}C NMR (75 MHz, $CDCl_3$) δ 143.24, 137.29, 129.86, 128.40, 127.23, 77.42, 54.58, 48.63, 35.63, 29.98, 21.69, 19.47.

HRMS calcd. For $C_{14}H_{20}NO_2S[M+H^+]$: 266.1215, found 266.1209.

(S)-7-methyl-1-tosyl-1,2,3,4,7,8-hexahydroazocine (4c):

The title compound was prepared from **2c** (34 μmol, 10.5 mg) following general procedure **E**. Purification by column chromatography (SiO_2, 1:8 Et_2O/Pentane, R_f (1:7 Et_2O/Pentane) = 0.37) afforded **4c** (77% yield, 7.0 mg, 98% *ee*, $[\alpha]_D$ = +5.7 (*c* 0.7, $CHCl_3$)) as a colourless oil.

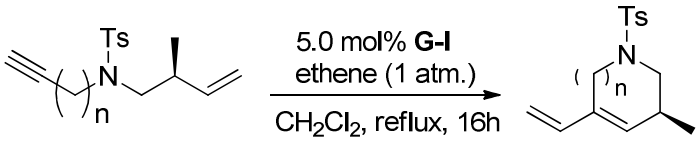
Enantiomeric excess determined by chiral HPLC analysis, Chiralpak OD-H (98% *n*-heptane/2% *i*-PrOH), 40 °C, retention times (min) 26.2 (major) and 27.6 (minor).

^{1}H NMR (200 MHz, $CDCl_3$) δ 7.68 (d, *J* = 8.3 Hz, 2H), 7.28 (d, *J* = 9.1 Hz, 2H), 5.70 – 5.54 (m, 1H), 5.40 – 5.30 (m, 1H), 3.54 – 3.43 (m, 1H), 3.36 (dt, *J* = 14.8 Hz, 4.1 Hz, 1H), 2.87 (ddd, *J* = 14.8 Hz, 10.7 Hz, 4.1 Hz, 1H), 2.75 – 2.63 (m, 1H), 2.47 –

2.34 (m, 2H), 2.47 – 2.31 (m, 5H), 2.10 – 1.99 (m, 2H), 1.52 – 1.39 (m, 1H), 1.01 (d, *J* = 6.9 Hz, 3H).
^{13}C NMR (100 MHz, $CDCl_3$) δ 142.85, 135.46, 129.57, 129.55, 126.84, 57.39, 48.42, 33.21, 29.84, 29.68, 24.09, 21.46, 18.80.
HRMS calcd. For $C_{15}H_{21}NO_2SNa[M+Na^+]$: 302.1202, found 302.1181

General procedure F: Ru-catalyzed ene-yne metathesis (8a-b).

Substrate (**6a-b**) was dissolved in degassed CH_2Cl_2 (5 mL) and Grubbs 1st generation catalyst (1.0 mol% per hour during 5 h) was added to the solution. The mixture was refluxed under an ethylene atmosphere (1 atm, balloon) until full conversion was reached, as judged by TLC. The mixture was concentrated under reduced pressure and purified by column chromatography to yield the desired products **8a**-**b** as a colourless oils.



n = 1,2,3 **6a-c** n = 1,2,3 **8a-c**

(S)-3-Methyl-1-tosyl-5-vinyl-1,2,3,6-tetrahydropyridine (8a):

The title compound was prepared from **6a** (0.15 mmol, 42 mg) following general procedure **F**. Purification by column chromatography (SiO_2, 1:9 Et_2O/Pet-Ether 40-60, R_f (5:95 Et_2O/Pet-Ether 40-60) = 0.15) afforded **8a** (77% yield, 31 mg, 99% *ee*, $[\alpha]_D$ = +33.6 (*c* 0.6, $CHCl_3$)) as a colourless oil.
Enantiomeric excess determined by chiral HPLC analysis, Chiralpak AS-H (99% *n*-heptane/1% *i*-PrOH), 40 °C, retention times (min) 29.6 (minor) and 31.3 (major).
^{1}H NMR (300 MHz, $CDCl_3$) δ 7.70 (d, *J* = 8.2 Hz, 2H), 7.33 (d, *J* = 8.0 Hz, 2H), 6.24 (dd, *J* = 17.8 Hz, 11.0 Hz, 1H), 5.63 (s, 1H), 5.11 – 4.93 (m, 2H), 3.88 (d, *J* = 15.4 Hz, 1H), 3.51 – 3.46 (m, 2H), 2.52 – 2.46 (m, 2H), 2.43 (s, 3H), 1.02 (d, *J* = 6.7 Hz, 3H).
^{13}C NMR (75 MHz, $CDCl_3$) δ 143.71, 136.57, 132.97, 131.59, 129.89, 127.85, 118.71, 111.91, 49.65, 44.26, 30.79, 21.71, 18.36.
HRMS calcd. For $C_{15}H_{20}NNaO_2S[M+Na^+]$: 300.1046, found 300.1026

(S)-3-Methyl-1-tosyl-5-vinyl-2,3,6,7-tetrahydro-1H-azepine (8b):

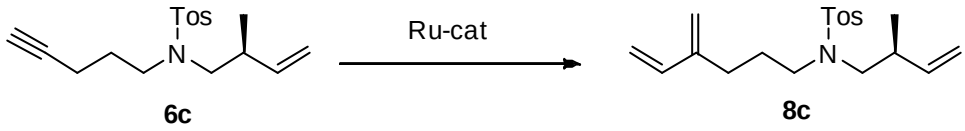
The title compound was prepared from **6b** (38 μmol, 11 mg) following general procedure **F**. Purification by column chromatography (SiO_2, 1:6 Et_2O/Pet-Ether 40-60, R_f (1:4 Et_2O/Pet-Ether 40-60) = 0.38) afforded **8b** (65% yield, 7 mg, 99% *ee*, $[\alpha]_D$ = -8.4 (*c* 1.0, $CHCl_3$)) as a colourless oil.
Enantiomeric excess determined by chiral HPLC analysis, Chiralpak OJ-H (95% *n*-heptane/5% *i*-PrOH), 40 °C, retention times (min) 22.5 (minor) and 25.4 (major).
^{1}H NMR (400 MHz, $CDCl_3$) δ 7.65 (d, *J* = 8.1 Hz, 2H), 7.29 (d, *J* = 8.4 Hz, 2H), 6.24 (dd, *J* = 17.5 Hz, 10.8 Hz, 1H), 5.55 (d, *J* = 3.4 Hz, 1H), 5.07 – 4.93 (m, 2H), 3.67 (ddd, *J* = 13.1 Hz, 8.0 Hz, 2.3 Hz, 1H), 3.60 – 3.53 (m, 1H), 2.89 (ddd, *J* = 13.0 Hz, 8.9 Hz, 1.9 Hz, 1H), 2.71 – 2.66 (m, 2H), 2.59 – 2.50 (m, 1H), 2.41 (s, 3H), 1.08 (d, *J* = 6.3 Hz, 3H).

^{13}C NMR (100 MHz, $CDCl_3$) δ 143.38, 140.37, 139.20, 138.82, 136.13, 129.90, 127.31, 111.41, 53.80, 47.10, 34.37, 27.55, 21.73, 19.98.
HRMS calcd. For $C_{16}H_{22}NO_2S[M+H^+]$: 292.1371, found 292.1366.

(S)-4-methyl-N-(2-methylbut-3-en-1-yl)-N-(4-methylenehex-5-en-1-yl)benzenesulfonamide (8c)

The title compound was prepared from **6c** (43 μmol, 13 mg) following general procedure **F** with the following modification: Grubbs 1st generation catalyst was added to the reaction mixture at 2.0 mol% each 8 h for a period of 7 days. Purification by column chromatography (SiO_2, 1:7 Et_2O/Pet-Ether 40-60, R_f = 0.42) afforded **8c** (35% yield, 5 mg, $[\alpha]_D$ = +4.0 (*c* 0.3, $CHCl_3$)) as a colourless oil.
^{1}H NMR (400 MHz, $CDCl_3$) δ 7.68 (d, *J* = 6.6 Hz, 2H), 7.27 (dd, *J* = 9.9, 4.7 Hz, 2H), 6.34 (dd, *J* = 17.0, 10.1 Hz, 1H), 5.67 (dd, *J* = 17.2, 7.4 Hz, 1H), 5.14 (d, *J* = 17.7 Hz, 1H), 5.09 – 4.88 (m, 5H), 3.10 (t, *J* = 10.3 Hz, 2H), 3.05 – 2.89 (m, 2H), 2.53 – 2.43 (m, 1H), 2.41 (s, 3H), 2.14 (t, *J* = 7.7 Hz, 2H), 1.70 (dd, *J* = 14.8, 6.9 Hz, 2H), 1.01 (d, *J* = 6.7 Hz, 2H).
^{13}C NMR (75 MHz, $CDCl_3$) δ 145.09, 143.00, 141.05, 138.59, 136.98, 129.56, 127.23, 116.00, 114.88, 113.39, 54.00, 48.63, 36.92, 28.50, 26.45, 21.46, 17.52.
HRMS calcd. For $C_{19}H_{28}NaNO_2S[M+Na^+]$: 370.1823, found 370.1801

Optimization for the preparation of 8c



6c **8c**

Entry	Catalyst	Solvent	atm	Temp.	Yield
1	**G-I** (5 mol%)	CH_2Cl_2	ethene	40 °C	9% [a,b]
2	**G-I** (10 mol%)	CH_2Cl_2	ethene	40 °C	35% [a,b]
3	**G-I** (5 mol%)	CH_2Cl_2	-	40 °C	n.d.[a]
4	**G-I** (5 mol%)	Toluene	ethene	70 °C	n.d.[a]
5	**G-I** (5 mol%)	Toluene	-	70 °C	n.d.[a]
6	**HG-II** (5 mol%)	CH_2Cl_2	ethene	40 °C	n.d.[a]
7	**HG-II** (5 mol%)	Toluene	ethene	70 °C	n.d.[a]
8	**G-II** (5 mol%)	CH_2Cl_2	ethene	40 °C	n.d.[a]

a Low conversion; *b* Complex mixture of products

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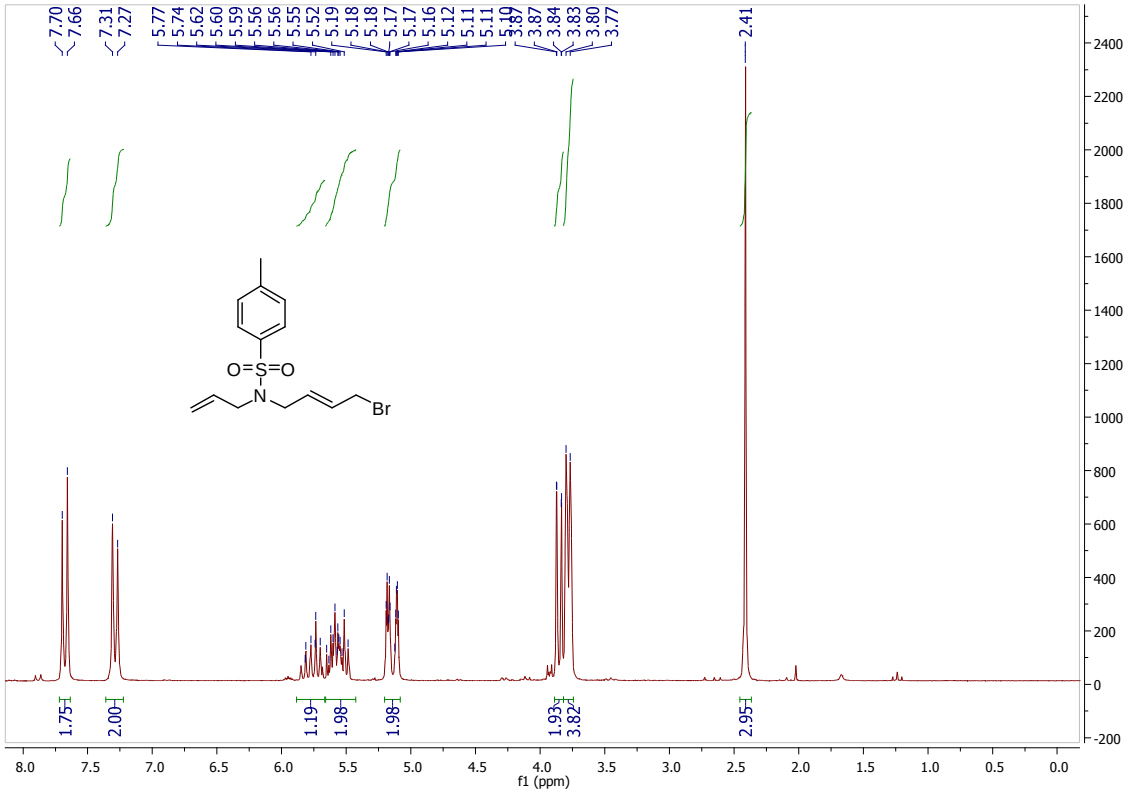
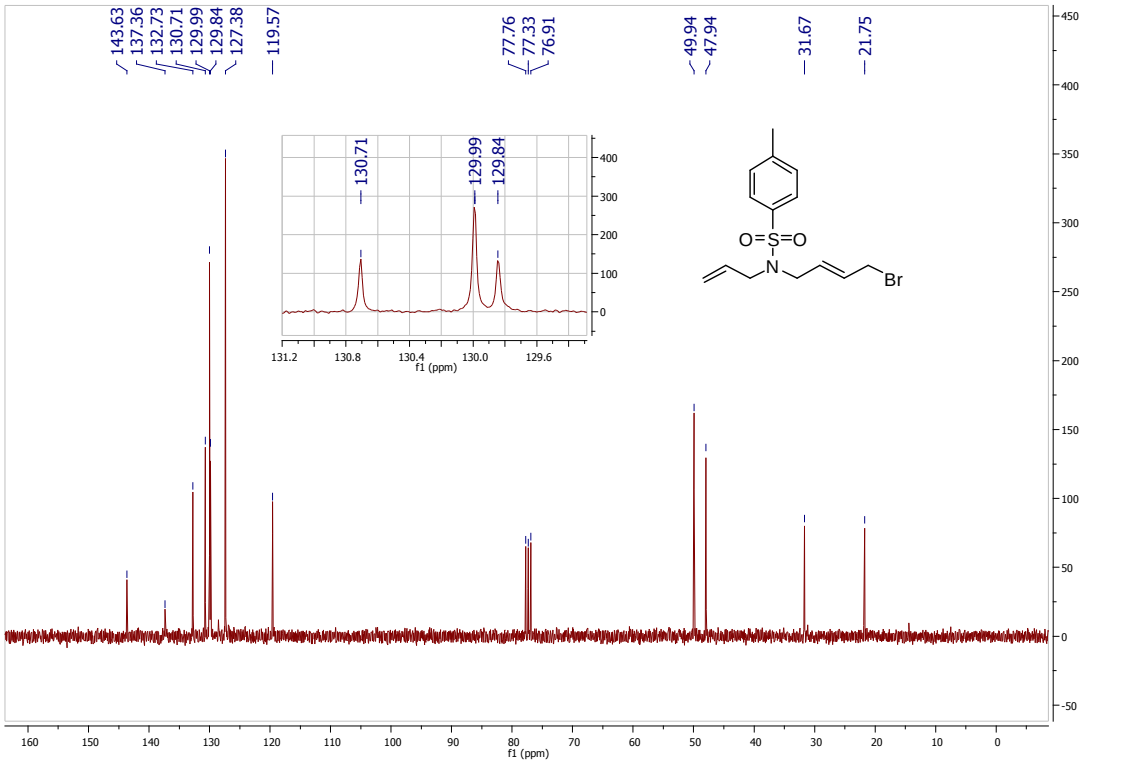
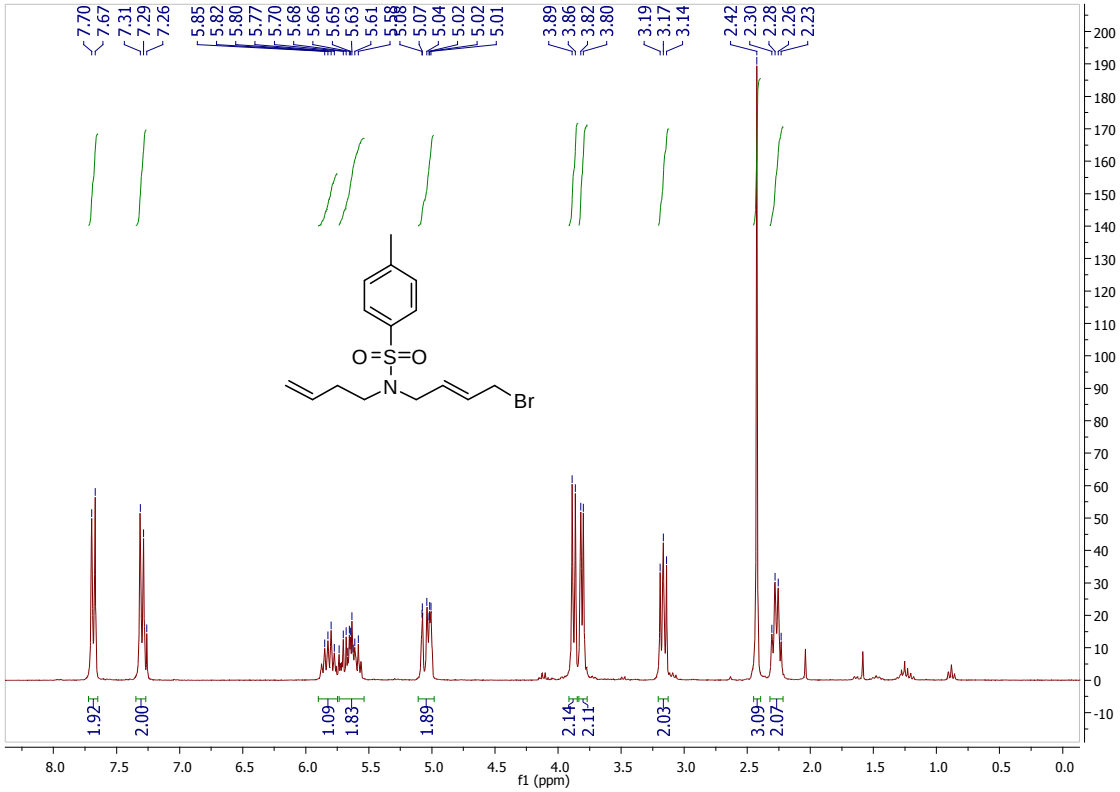
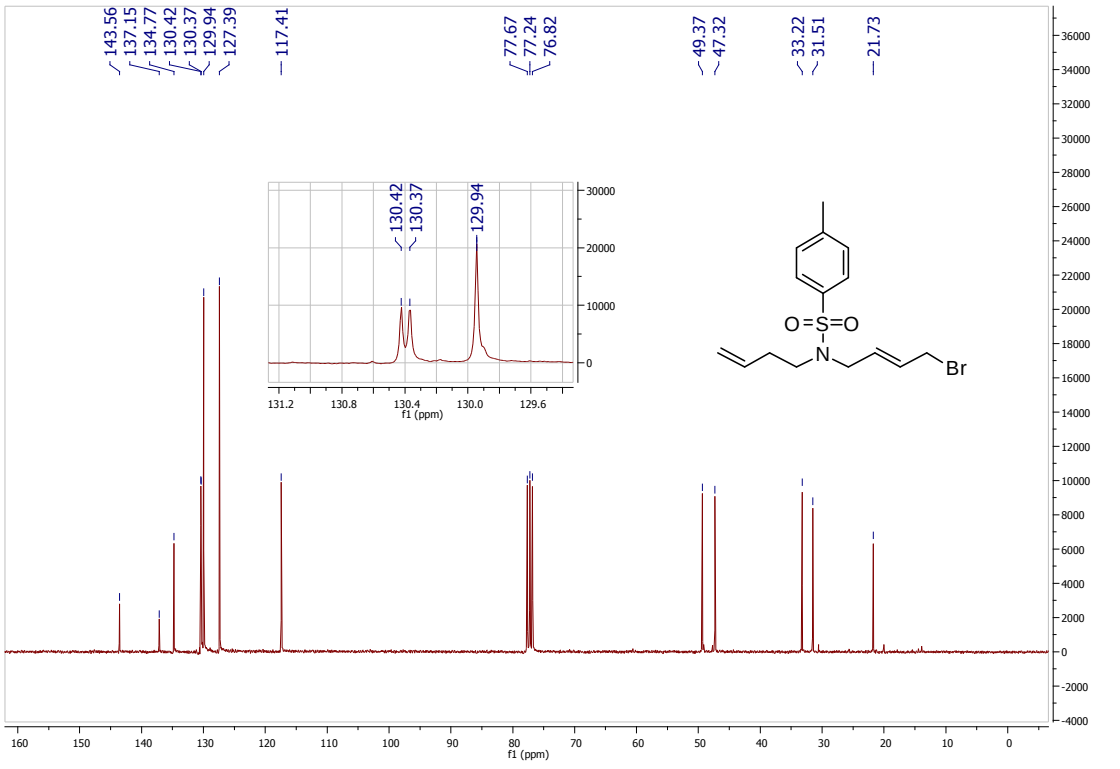
***(E)*-*N*-(4-bromo-2-buten-1-yl)-4-methyl-*N*-2-propen-1-yl-benzenesulfonamide (1a)**



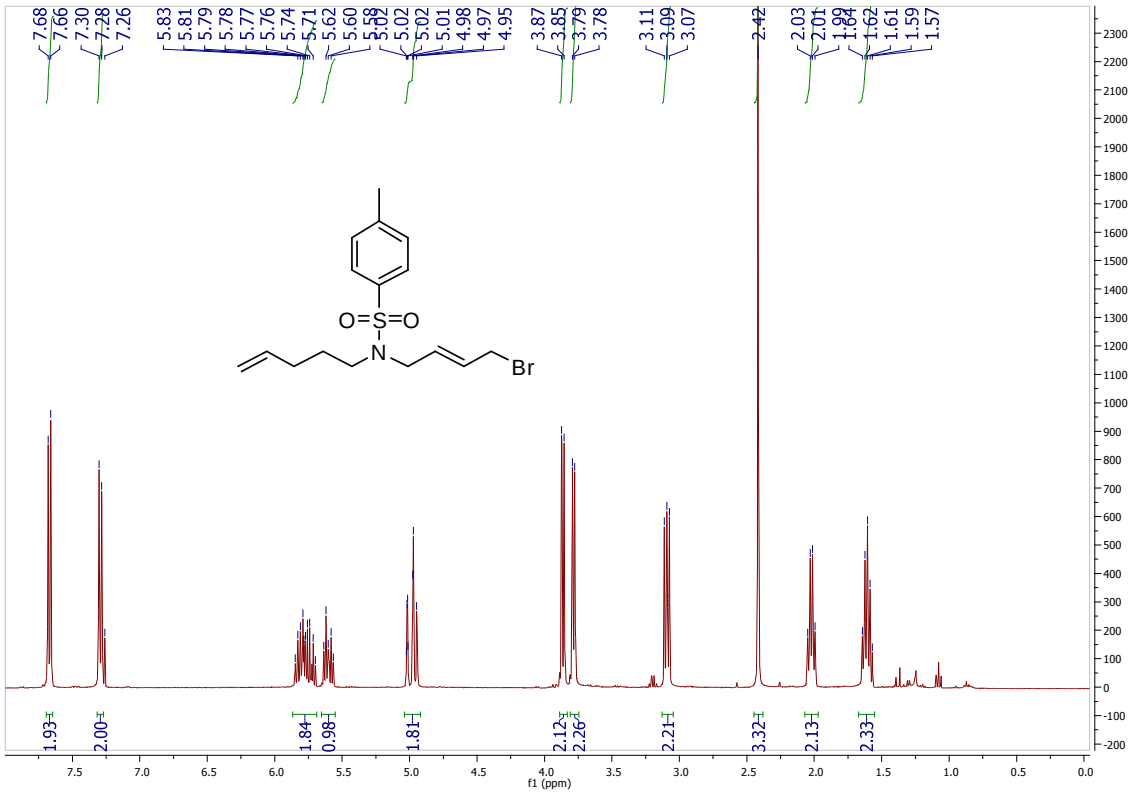


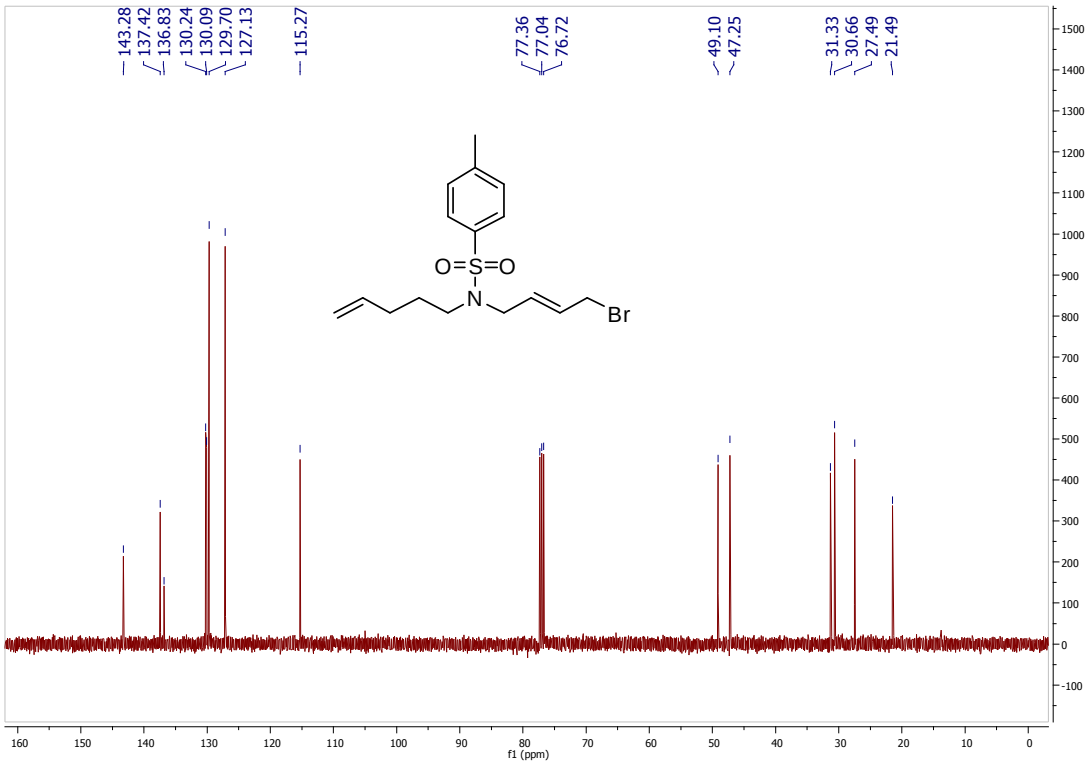
***(E)*-*N*-(4-bromo-2-buten-1-yl)-4-methyl-*N*-3-buten-1-yl-benzenesulfonamide (1b)**



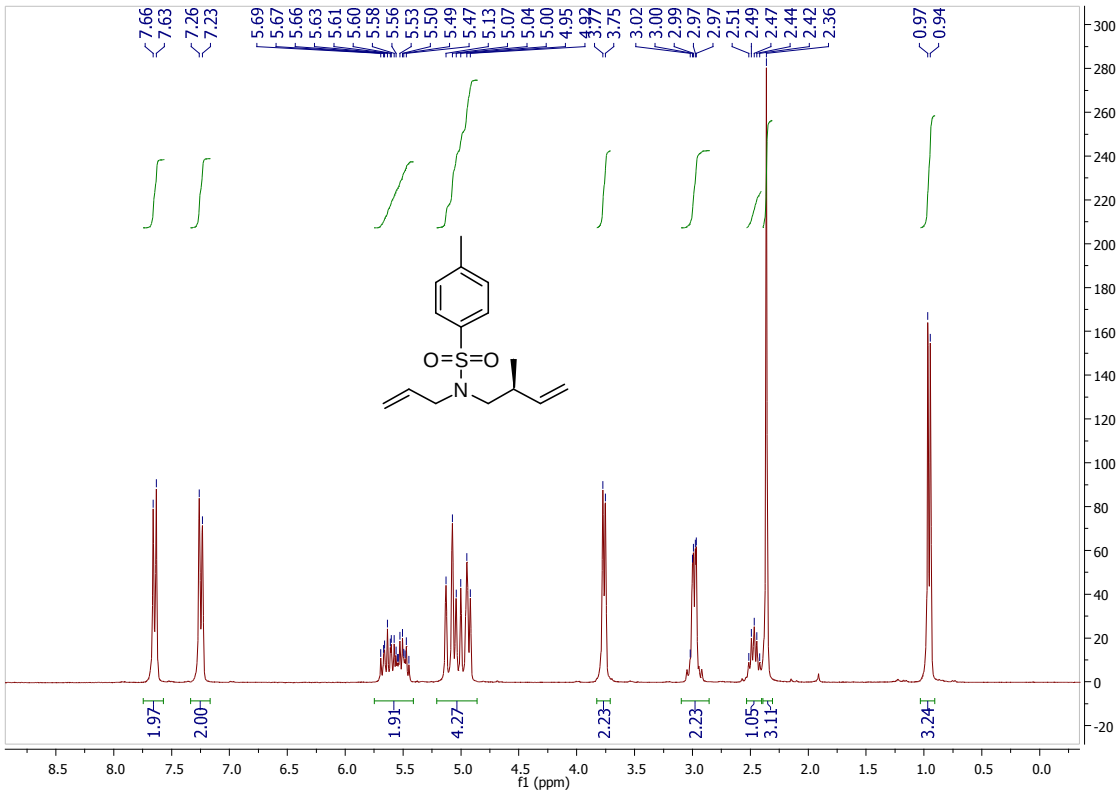


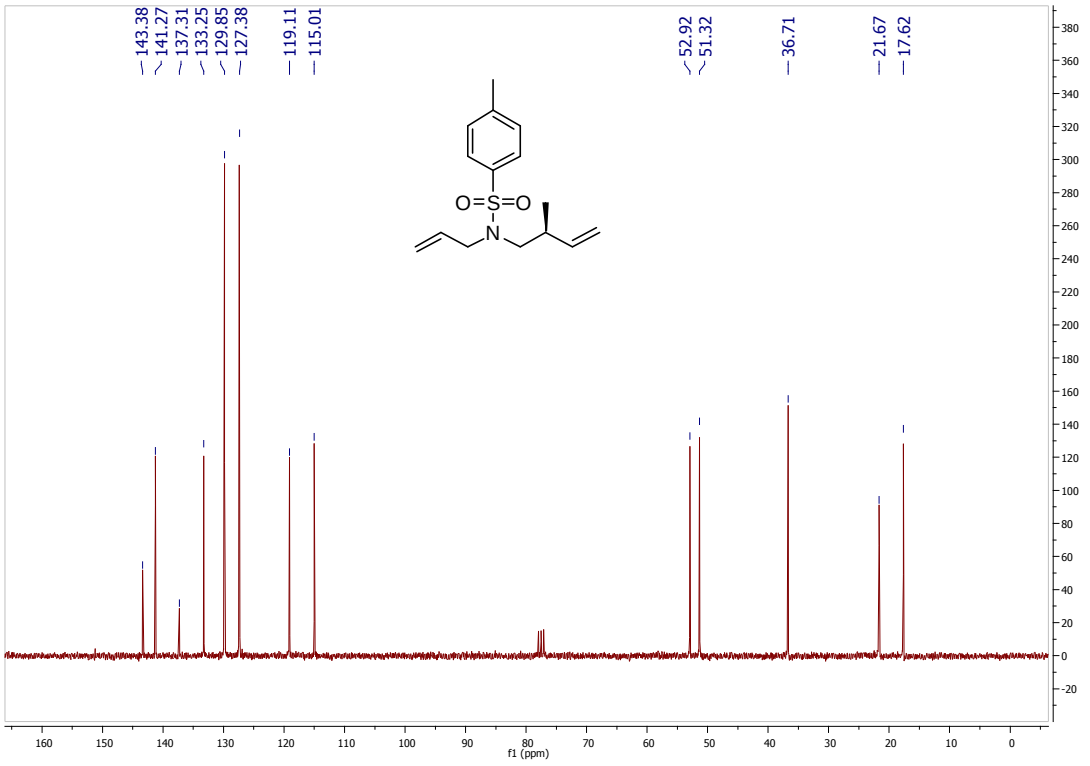
***(E)*-*N*-(4-bromo-2-buten-1-yl)-4-methyl-*N*-4-penten-1-yl-benzenesulfonamide (1c)**



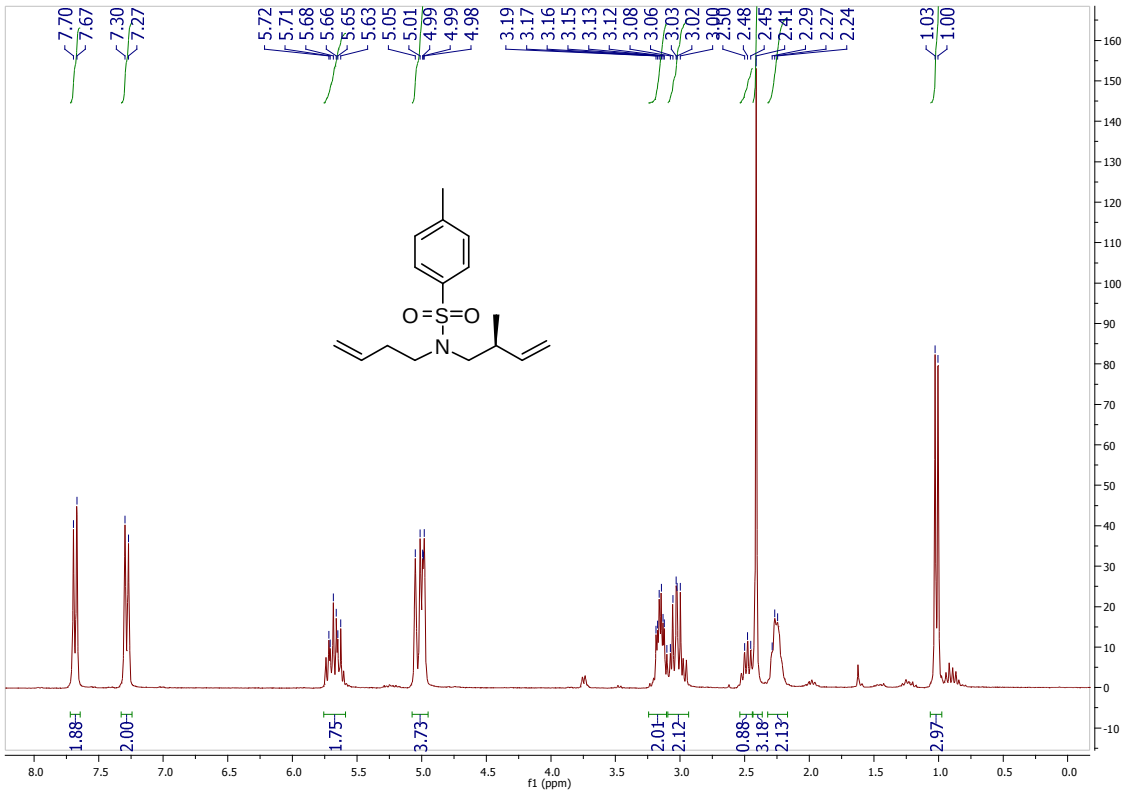


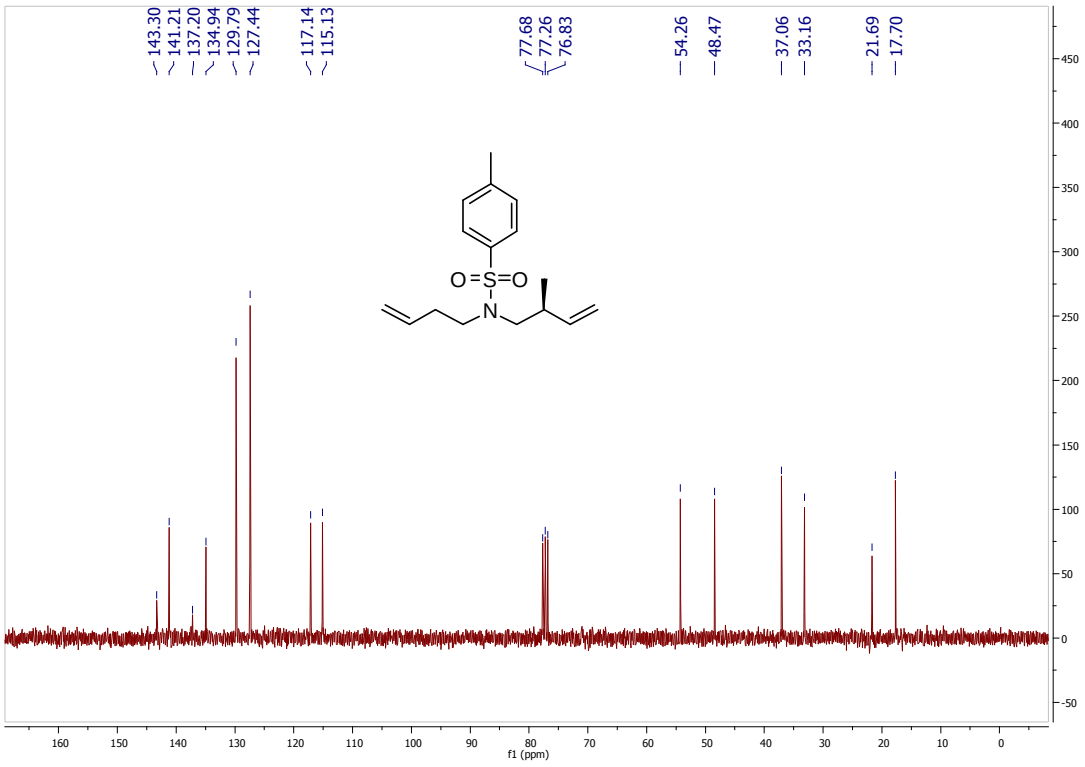
(S)-*N*-Allyl-4-methyl-*N*-(2-methylbut-3-en-1-yl)benzenesulfonamide (2a)



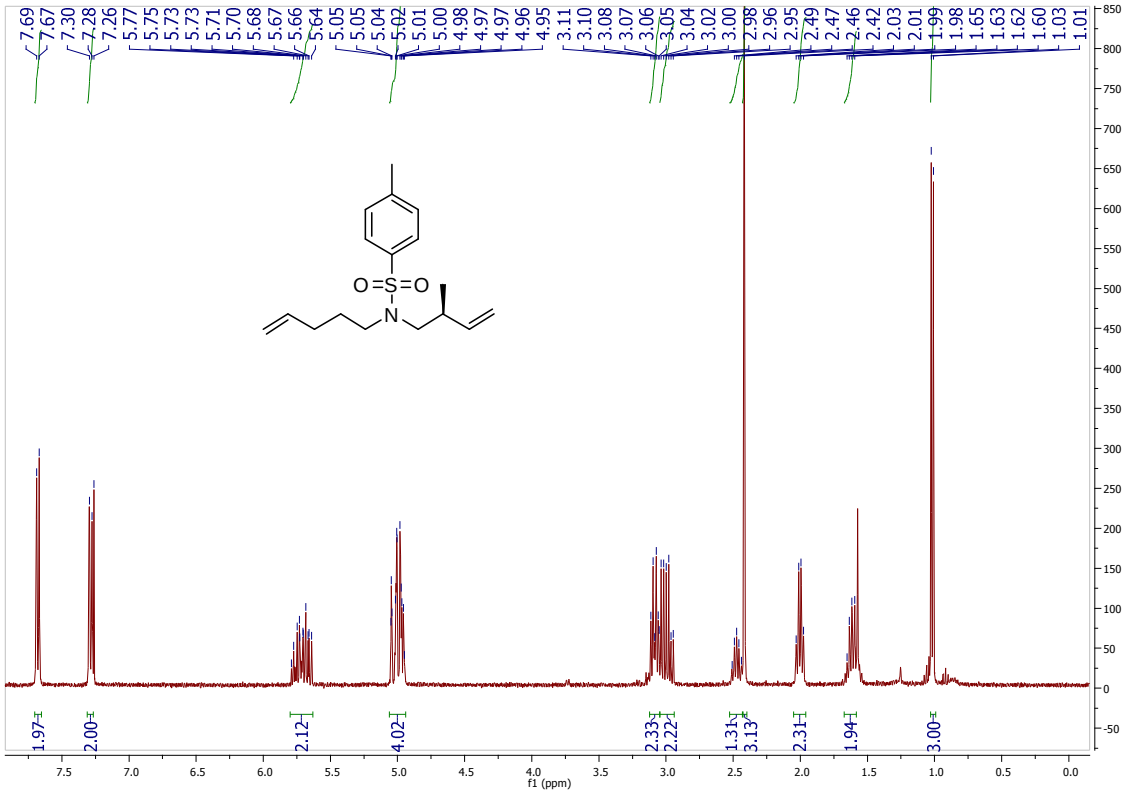


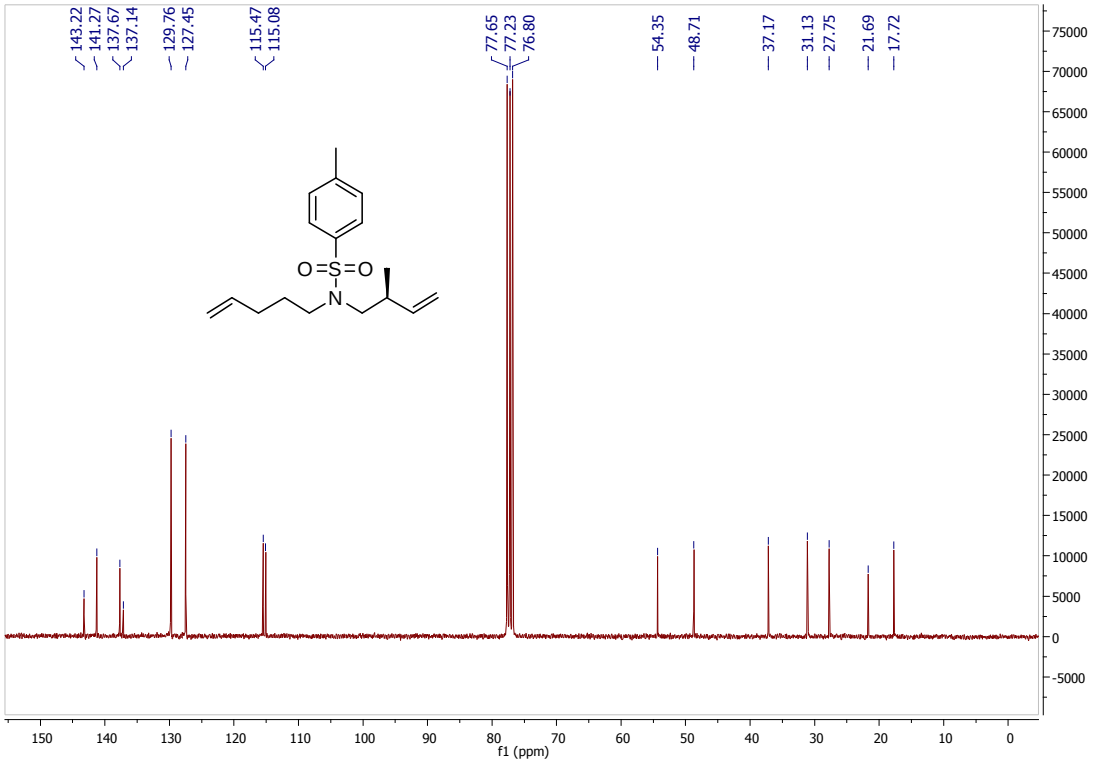
(S)-*N*-(but-3-en-1-yl)-4-methyl-*N*-(2-methylbut-3-en-1-yl)benzenesulfonamide (2b)



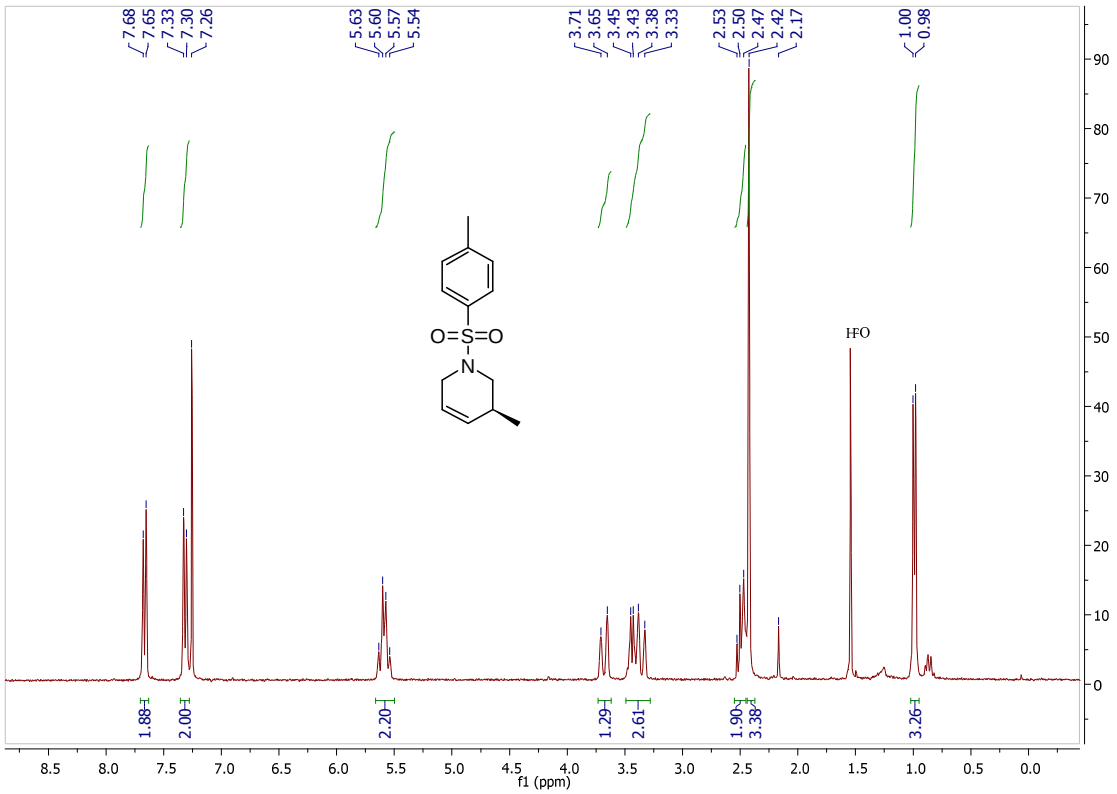


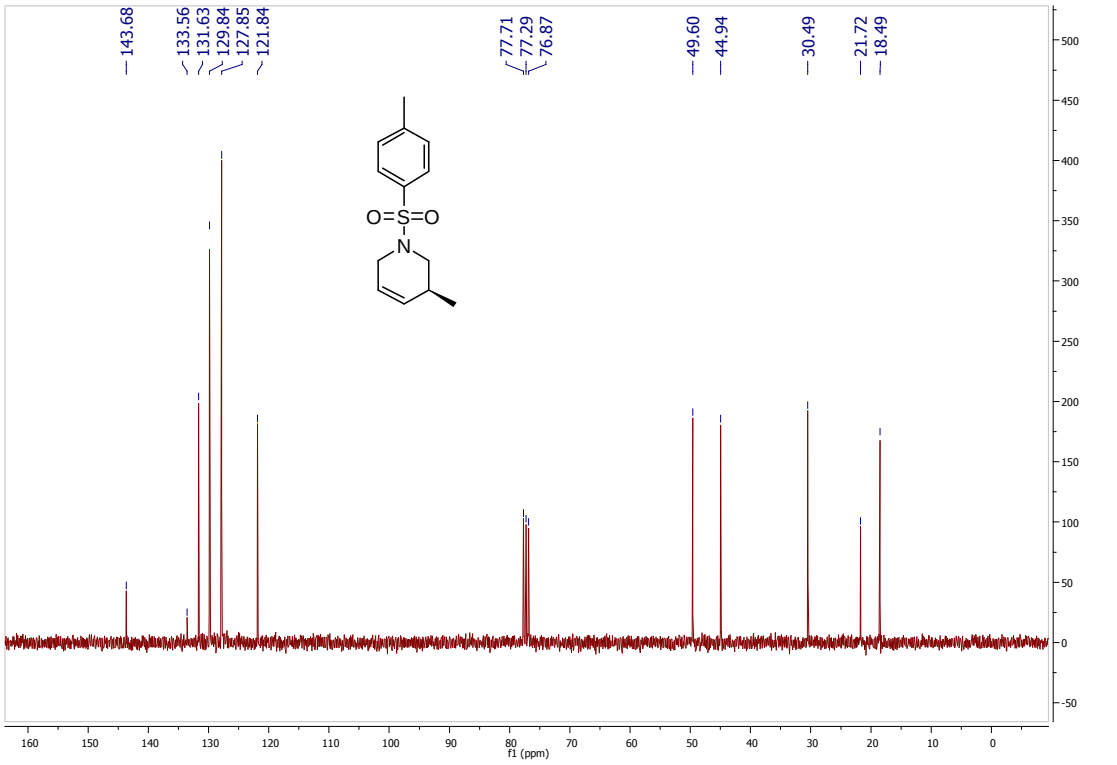
(S)-4-methyl-*N*-(2-methylbut-3-en-1-yl)-*N*-(pent-4-en-1-yl)benzenesulfonamide (2c)



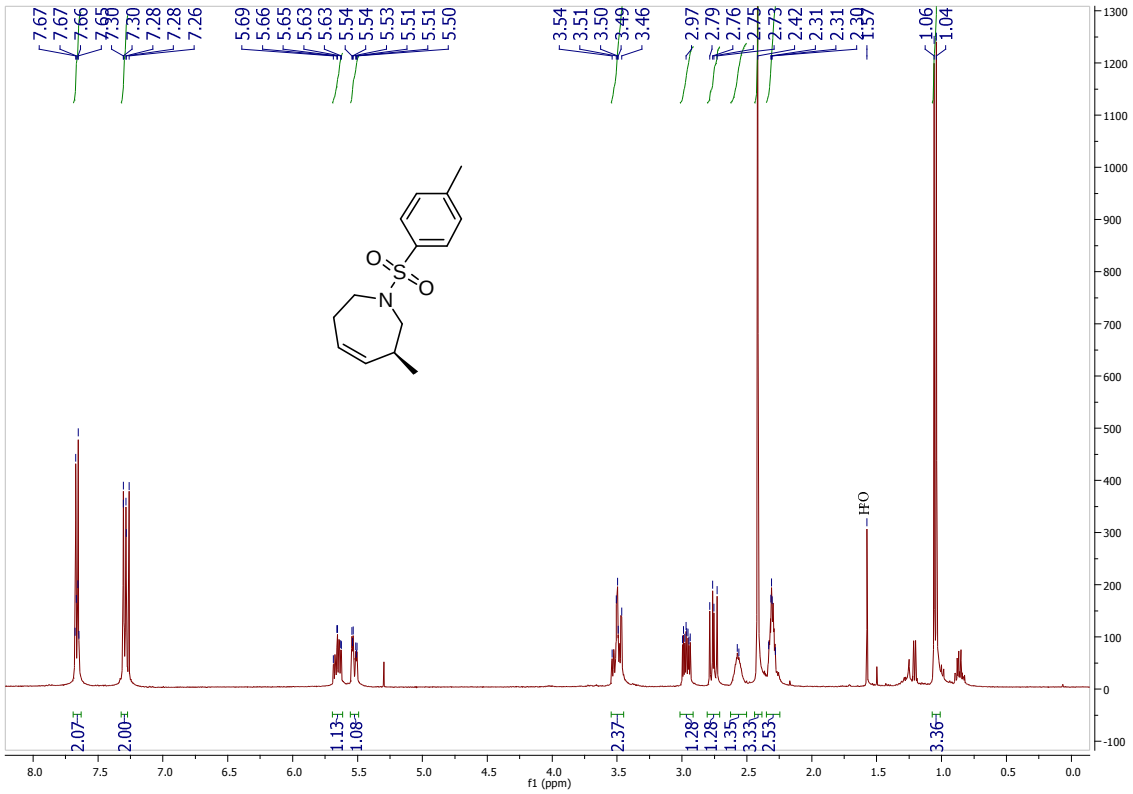


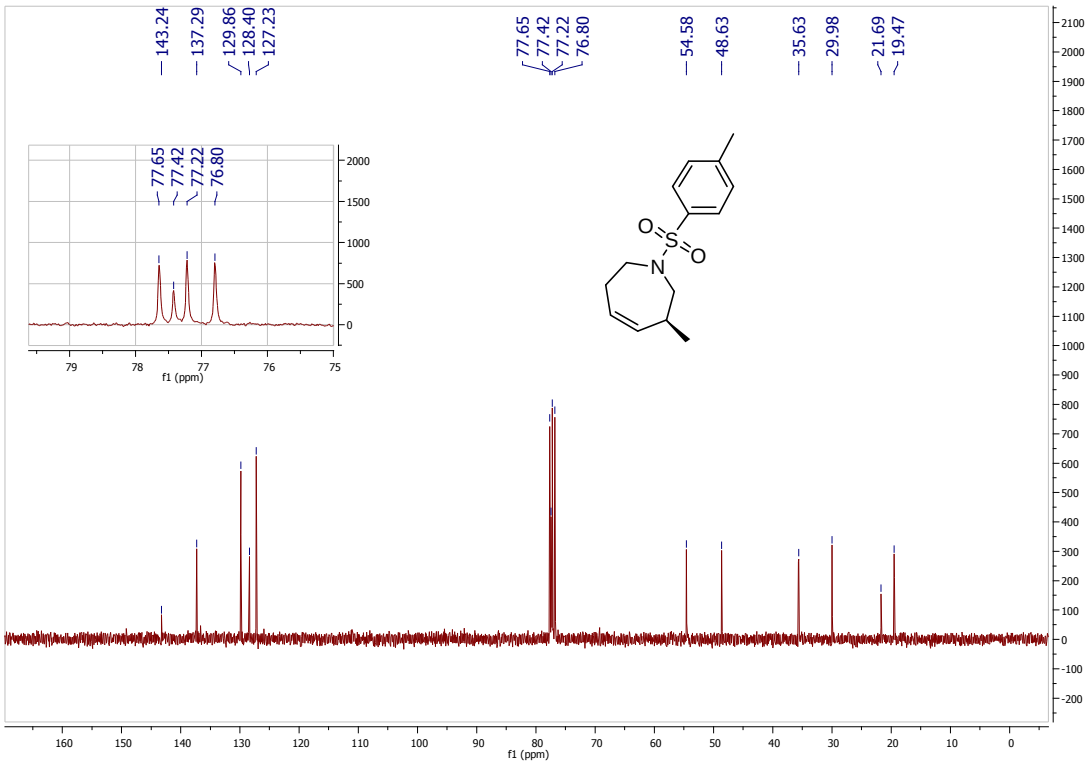
(S)-3-Methyl-1-tosyl-1,2,3,6-tetrahydropyridine (4a)



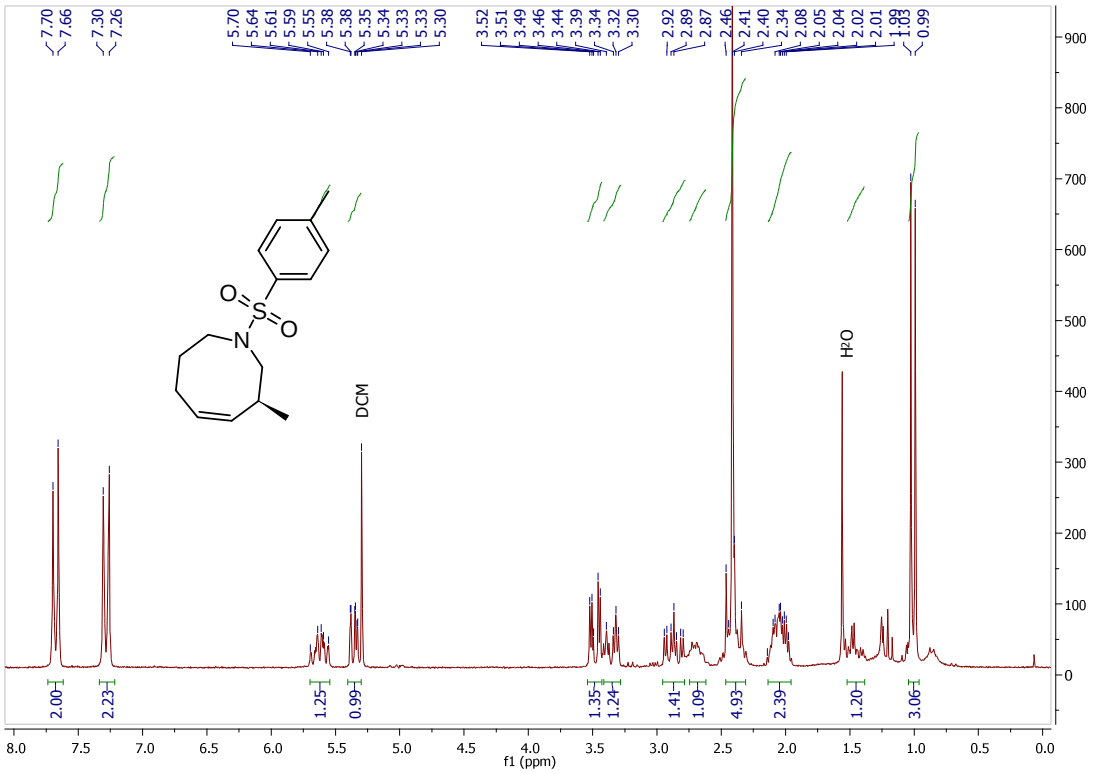


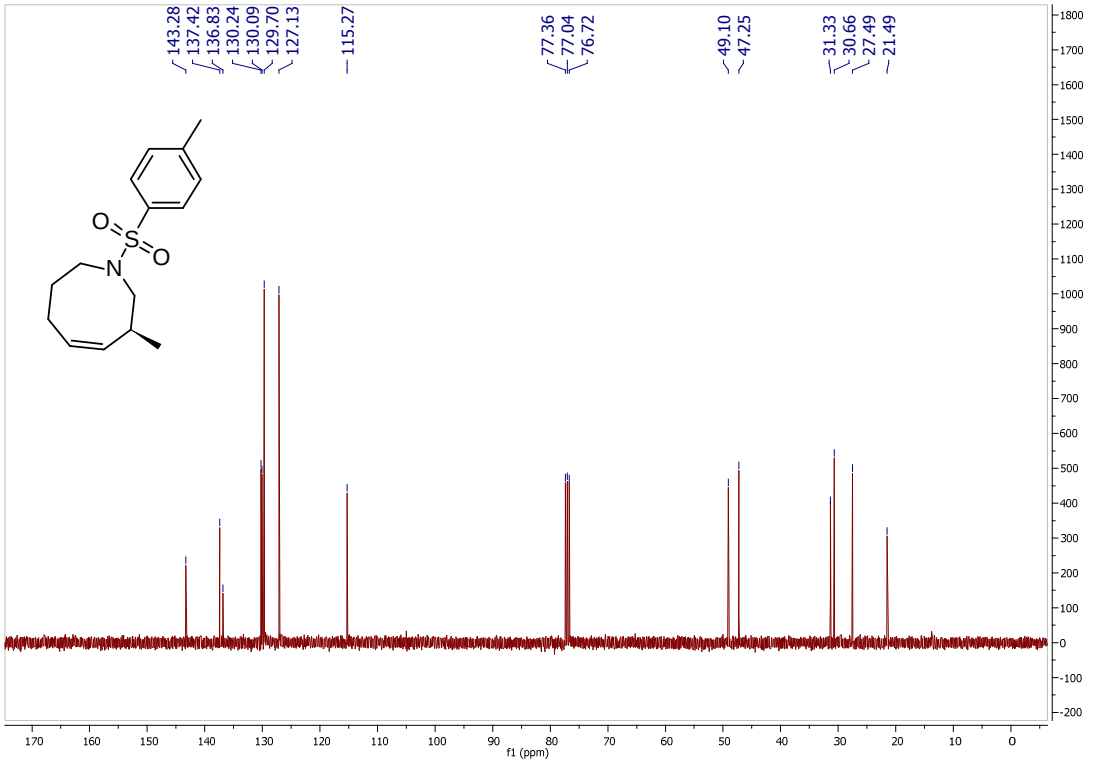
(S)-3-methyl-1-tosyl-2,3,6,7-tetrahydro-1H-azepine (4b)



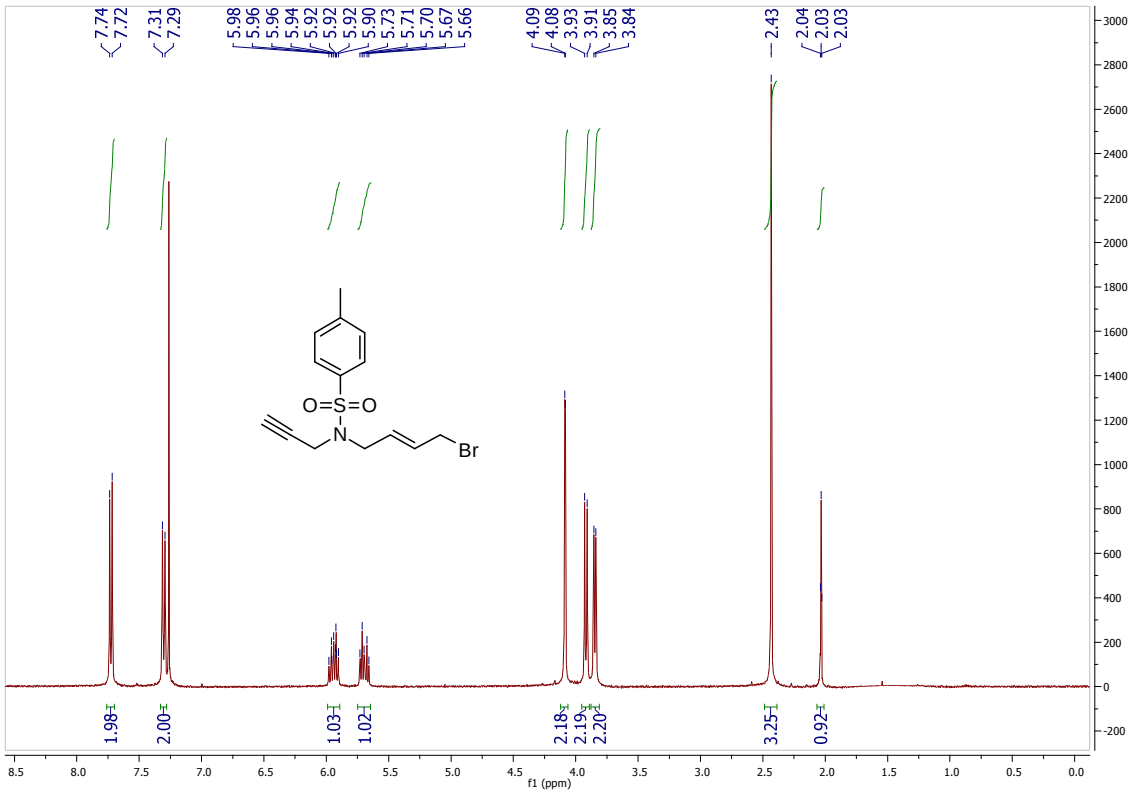


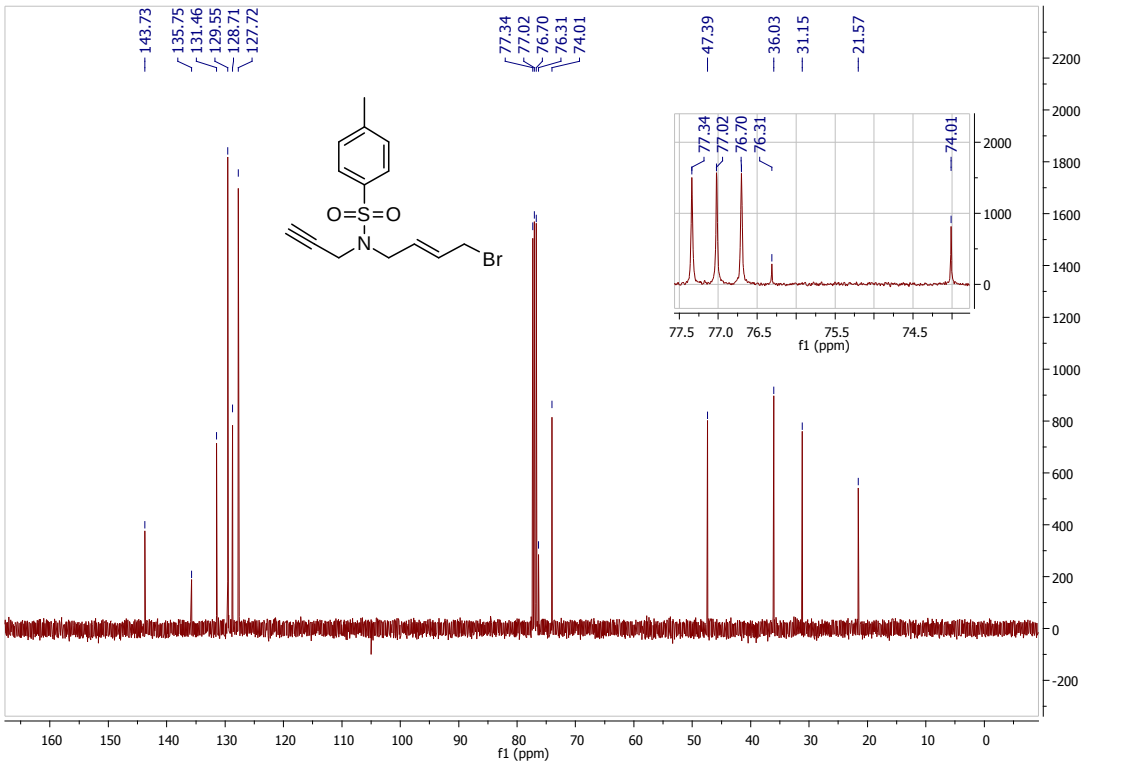
(S)-7-methyl-1-tosyl-1,2,3,4,7,8-hexahydroazocine (4c)



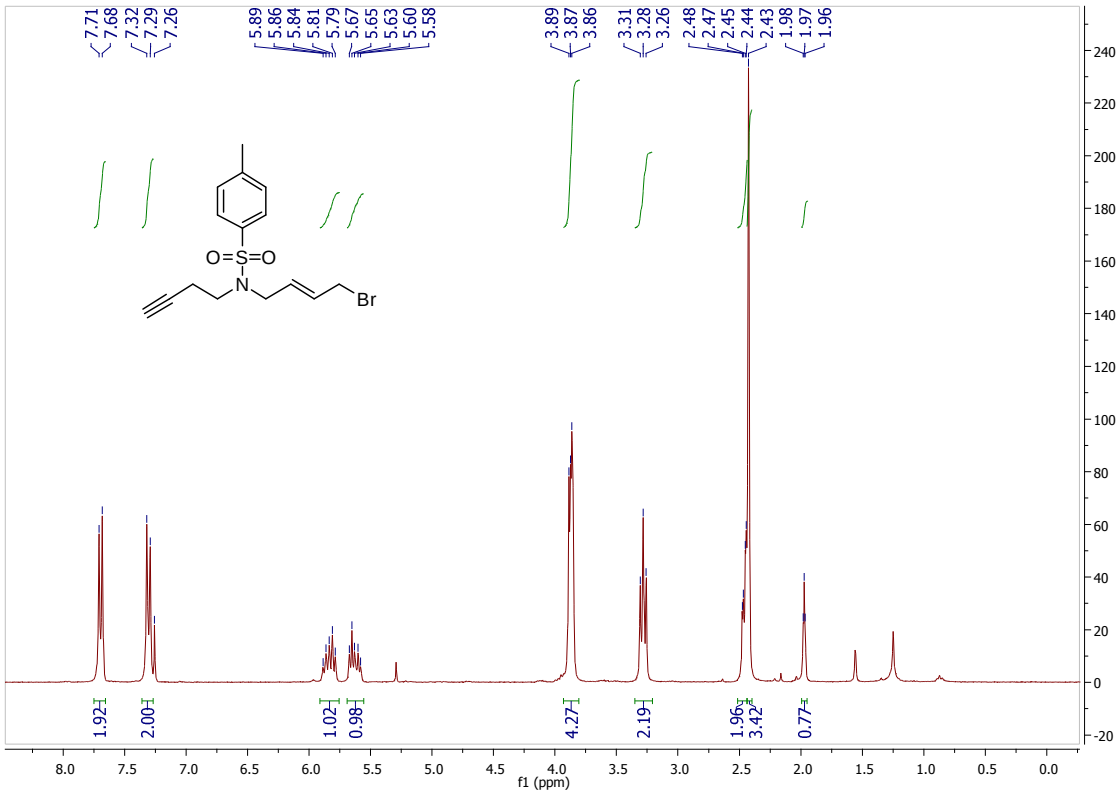


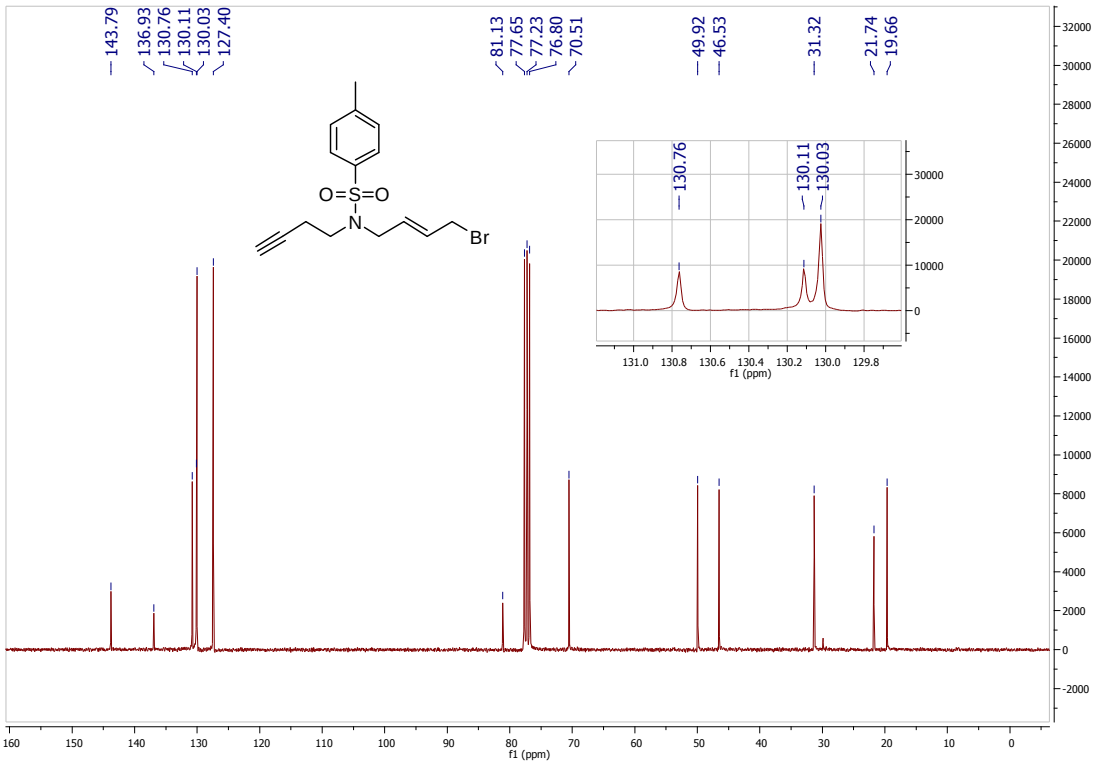
***(E)*-*N*-(4-bromo-2-buten-1-yl)-4-methyl-*N*-2-propyn-1-yl-benzenesulfonamide (5a)**



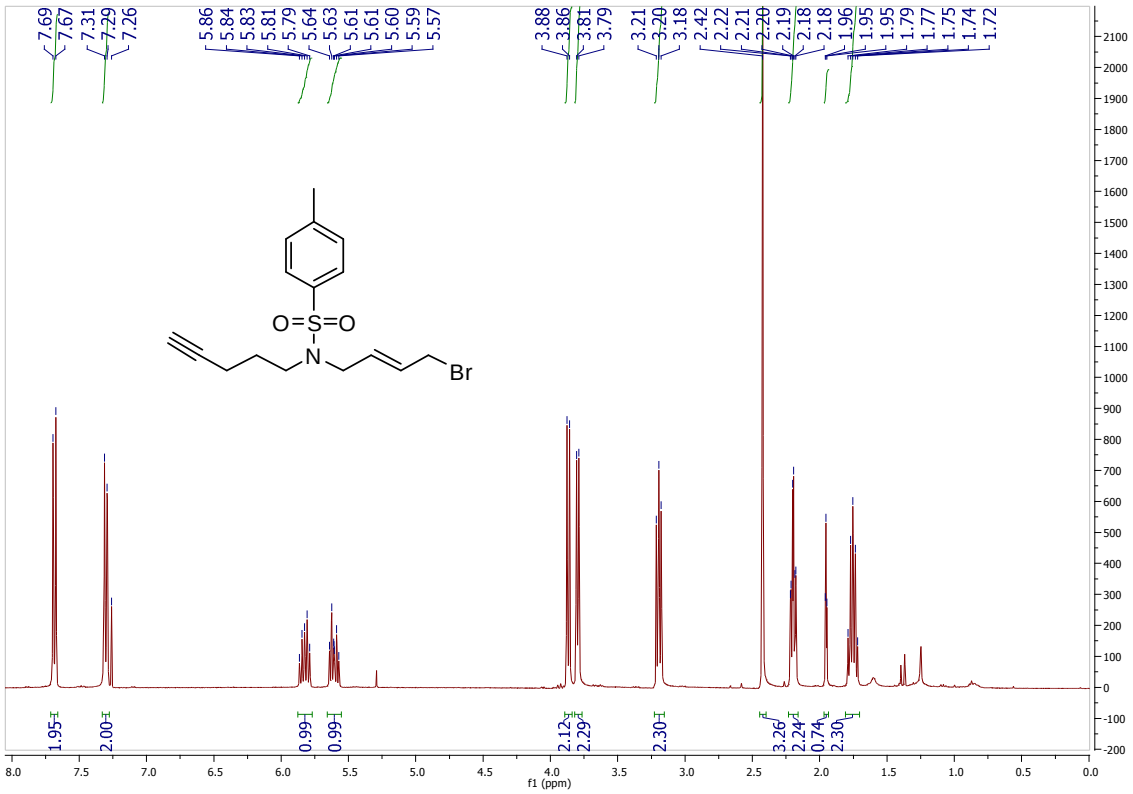


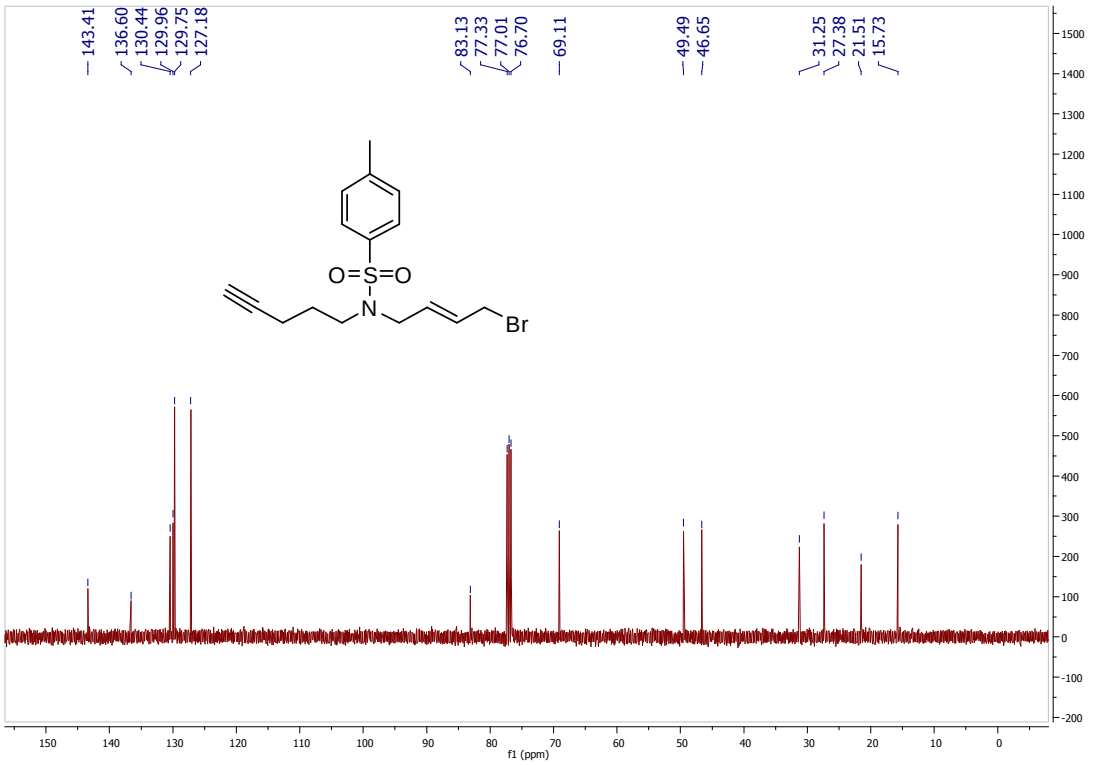
***(E)*-*N*-(4-bromo-2-buten-1-yl)-4-methyl-*N*-3-butyn-1-yl-benzenesulfonamide (5b)**



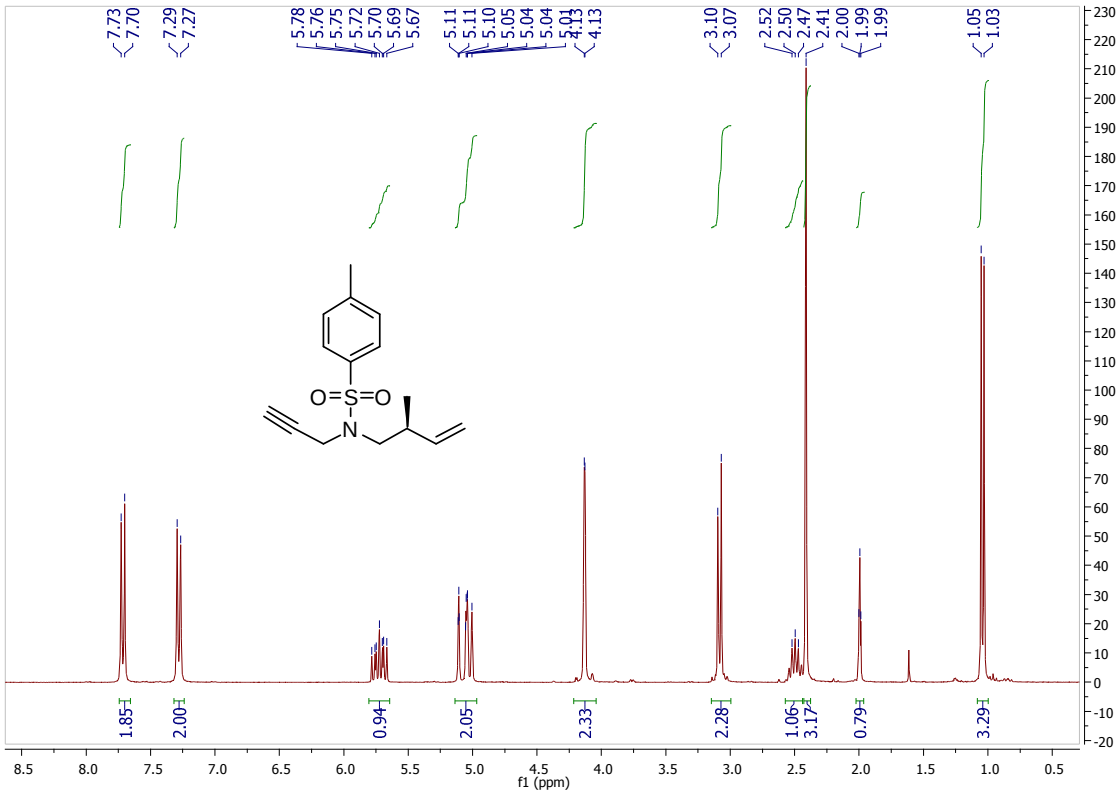


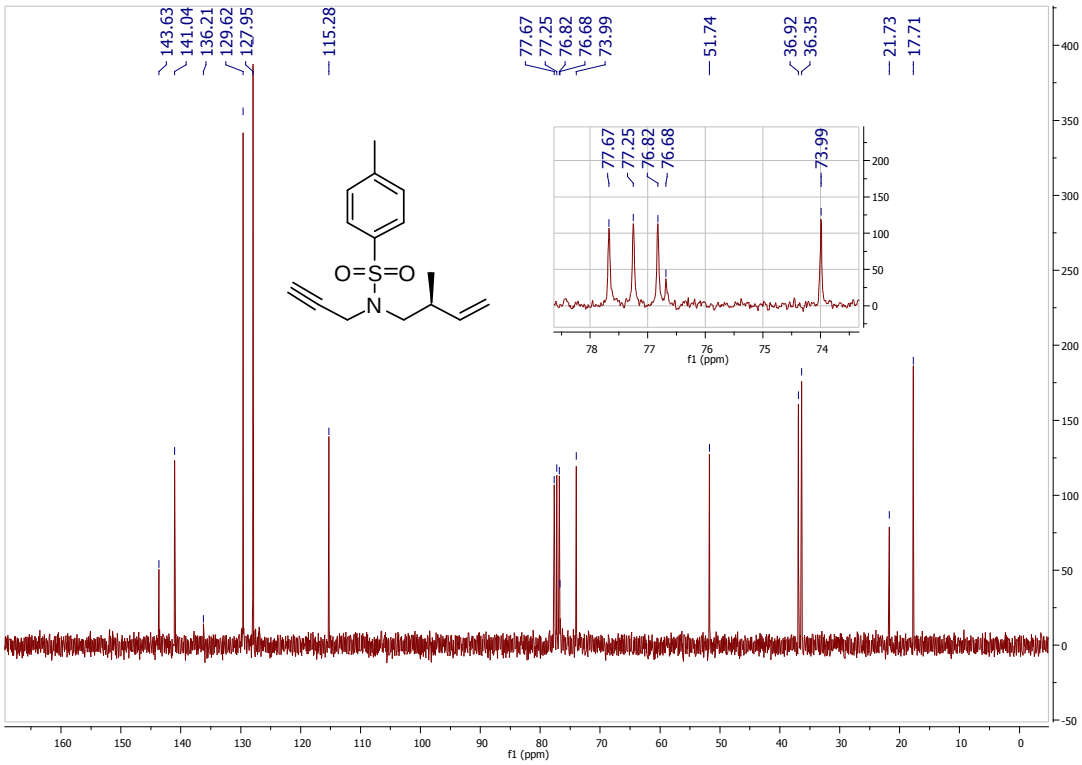
***(E)*-*N*-(4-bromo-2-buten-1-yl)-4-methyl-*N*-4-pentyn-1-yl-benzenesulfonamide (5c)**



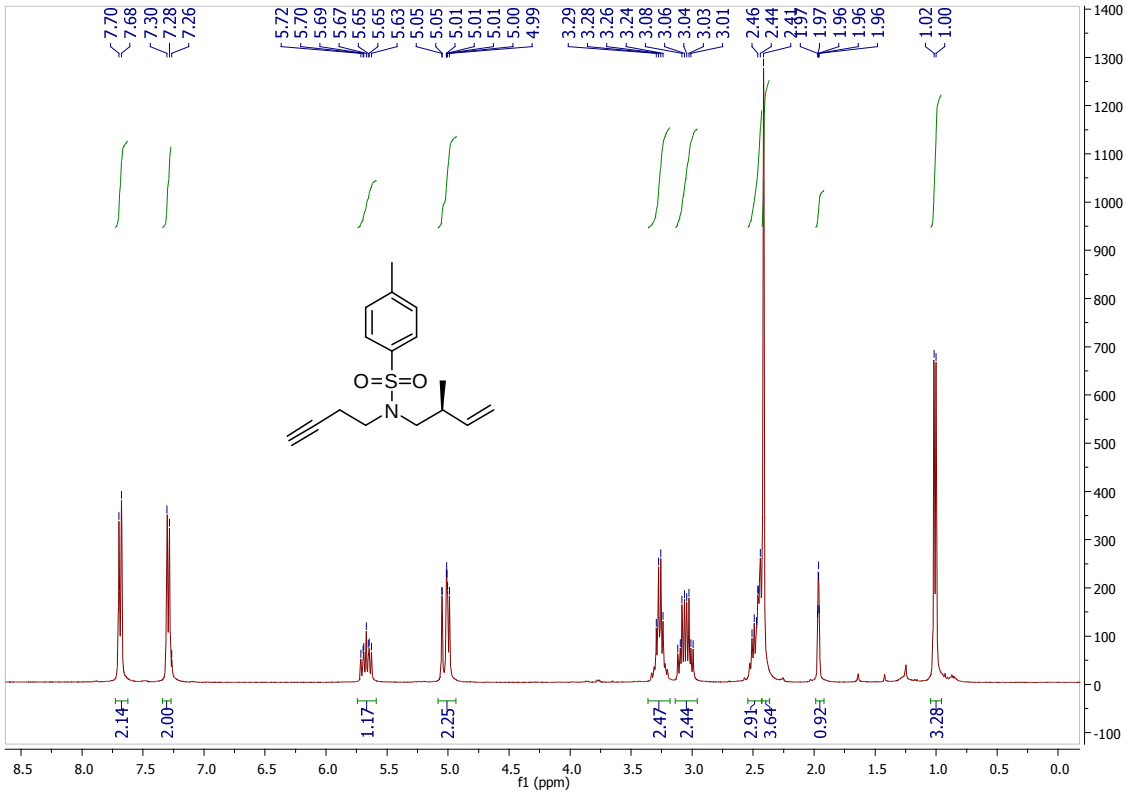


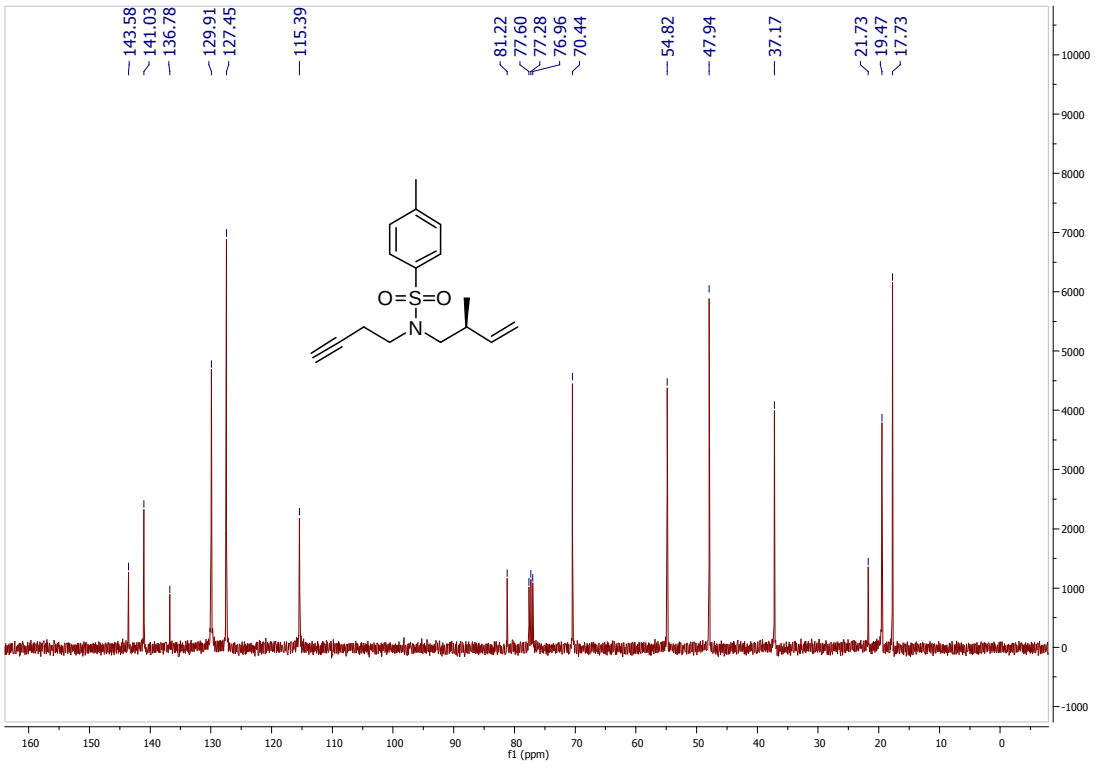
(S)-4-methyl-*N*-(2-methylbut-3-en-1-yl)-*N*-(prop-2-yn-1-yl)benzenesulfonamide (6a)





(S)-*N*-(but-3-yn-1-yl)-4-methyl-*N*-(2-methylbut-3-en-1-yl)benzenesulfonamide (6b)





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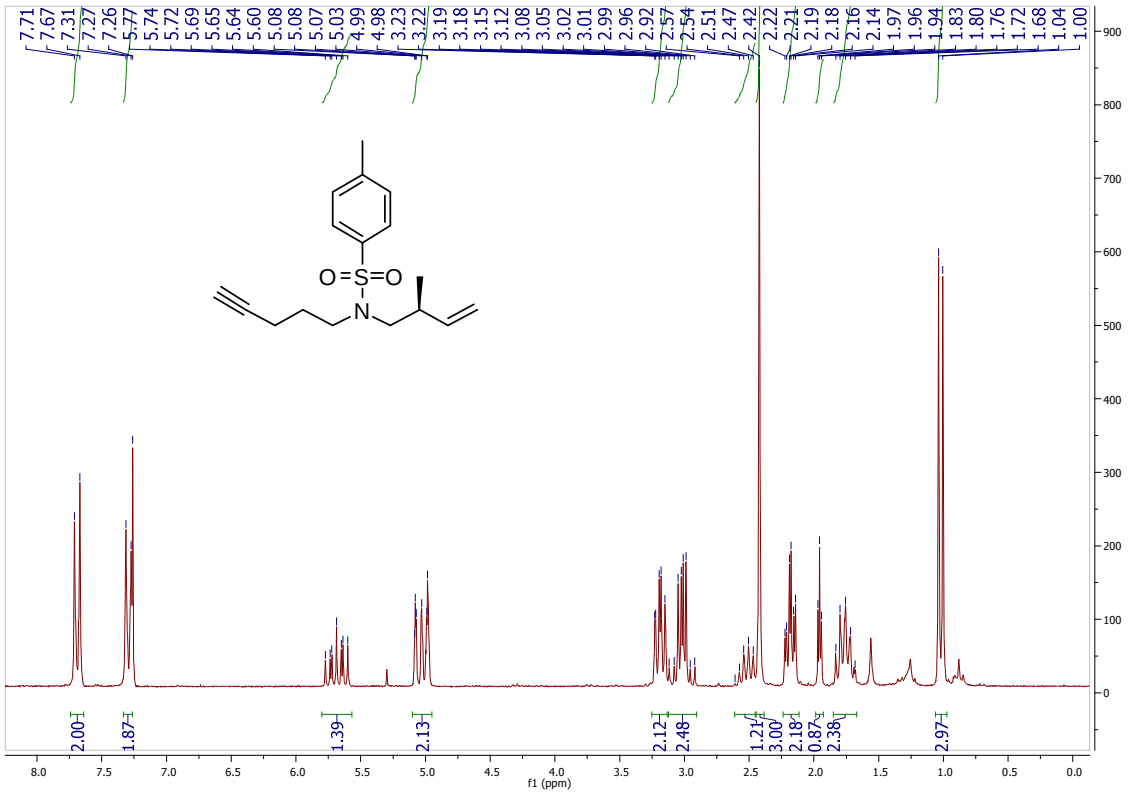
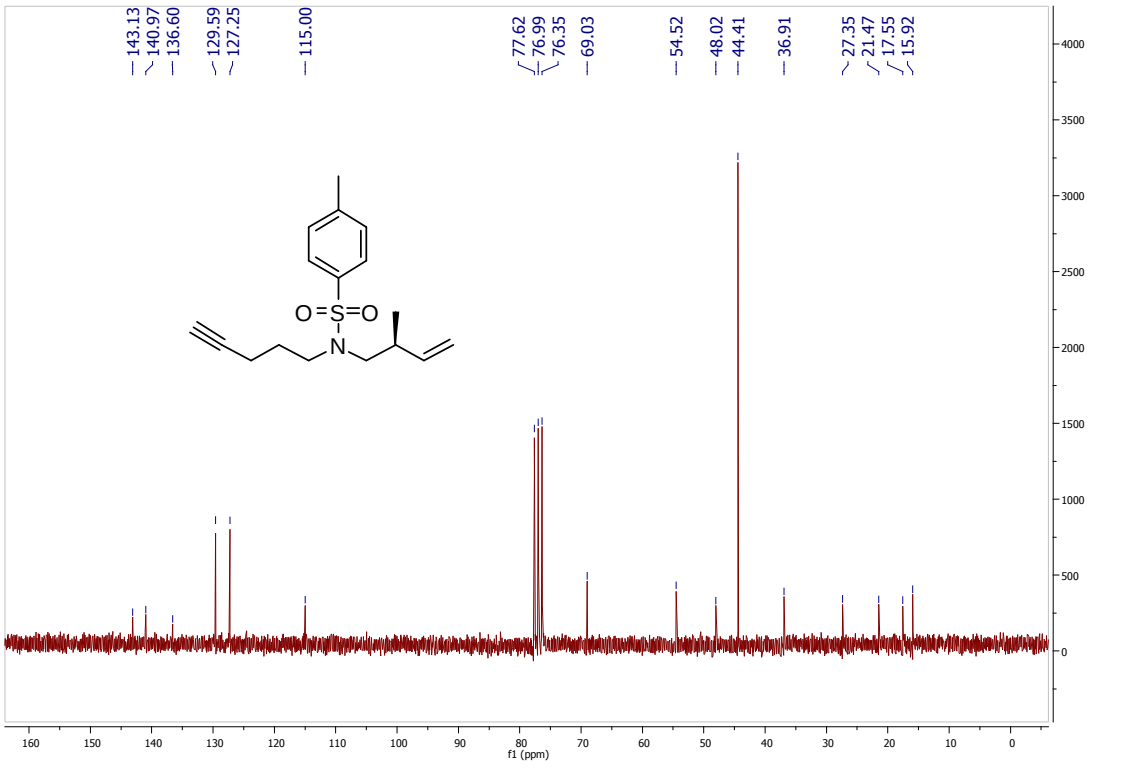
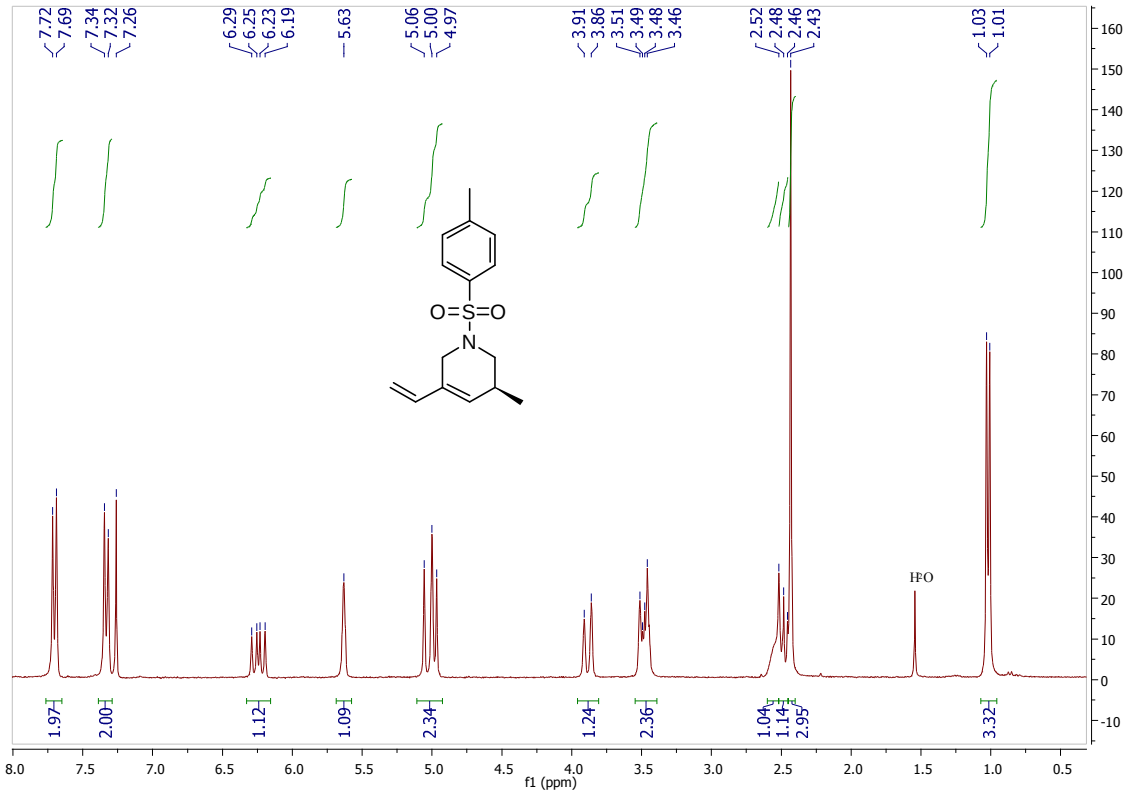
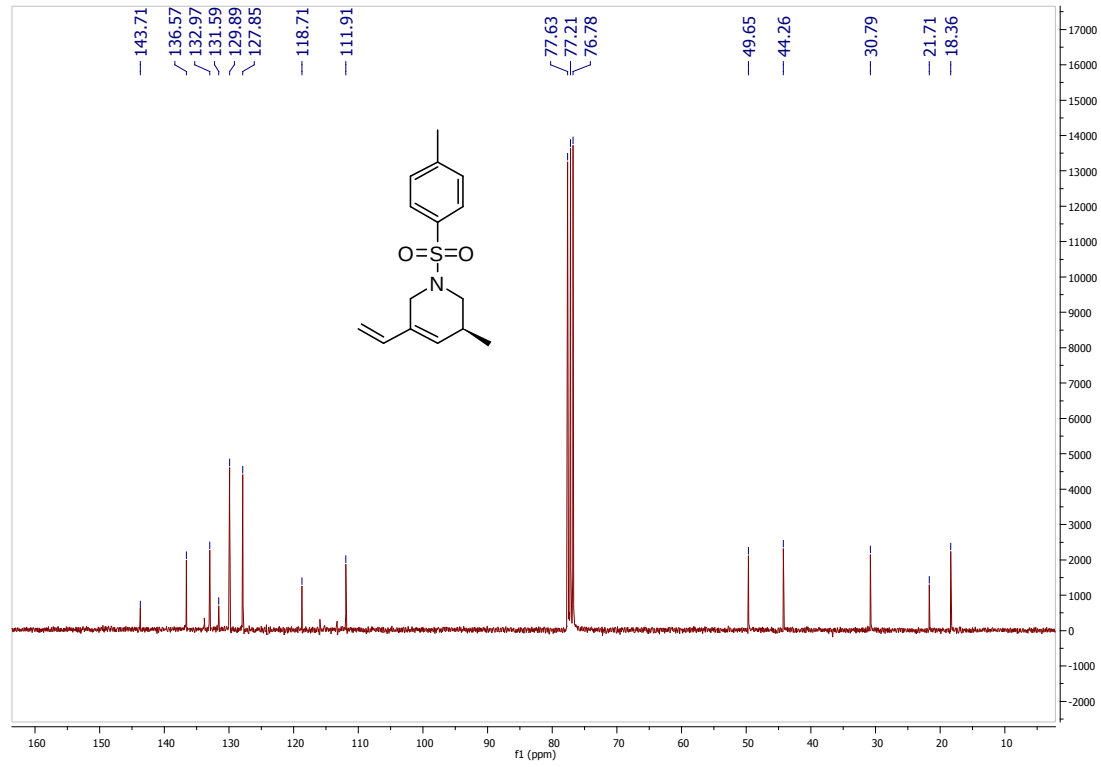


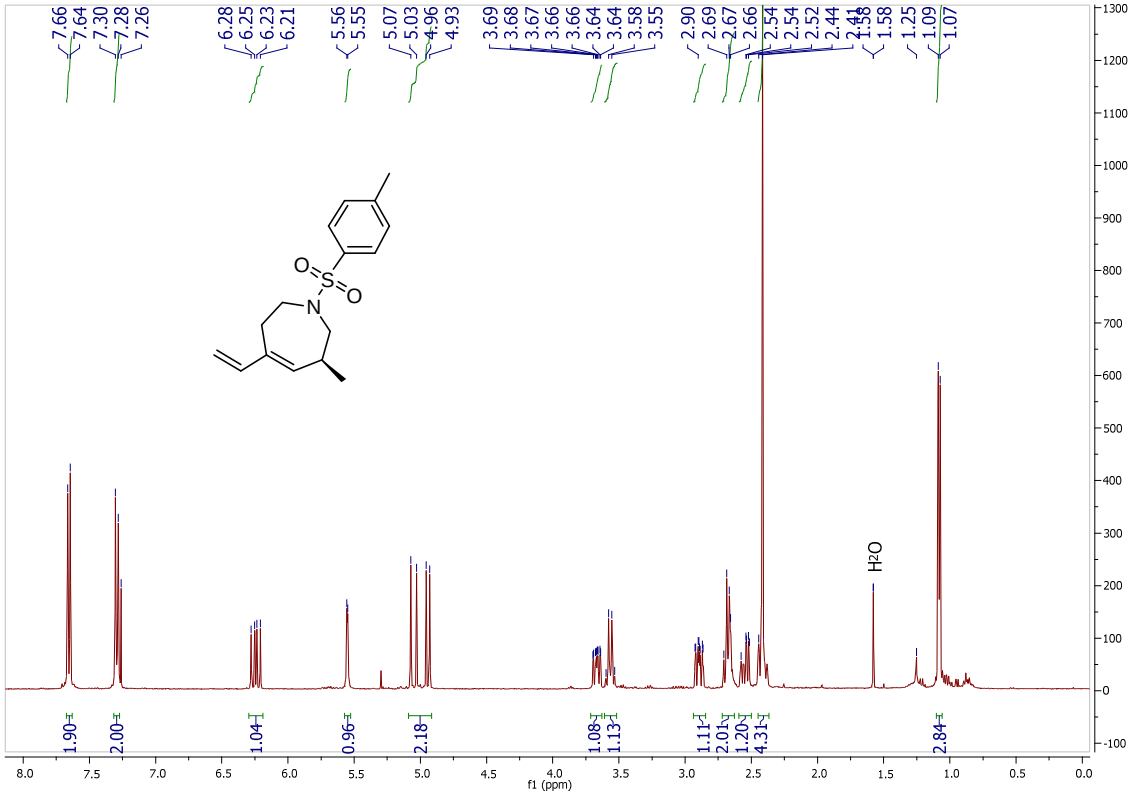


(S)-3-Methyl-1-tosyl-5-vinyl-1,2,3,6-tetrahydropyridine (8a)





(S)-3-Methyl-1-tosyl-5-vinyl-2,3,6,7-tetrahydro-1H-azepine (8b)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1.09
1.07
H2O
1.90
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1.13
1.11
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1.20
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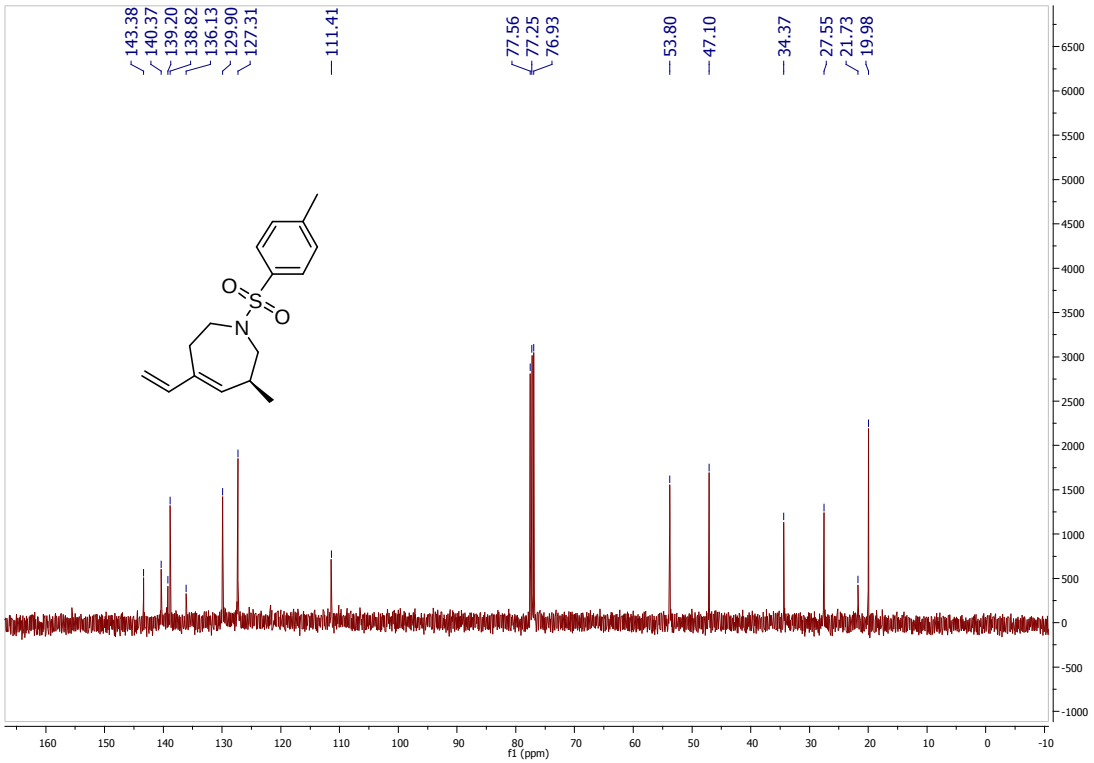
2.84
f1 (ppm)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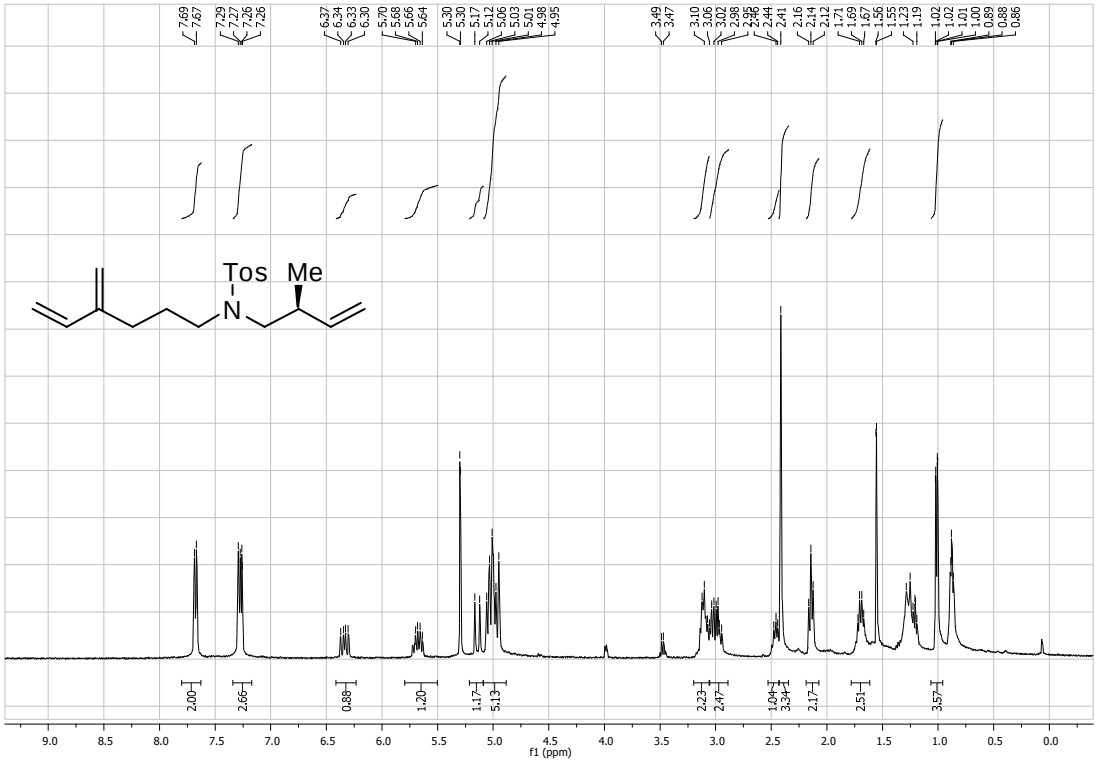
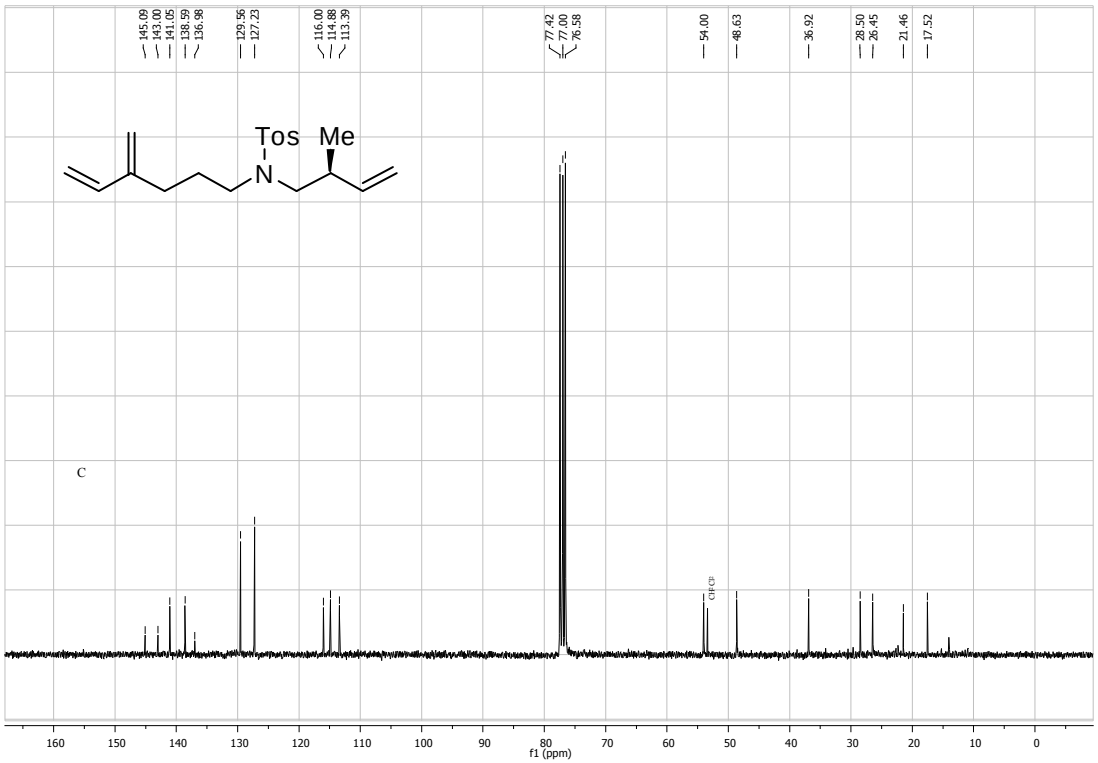
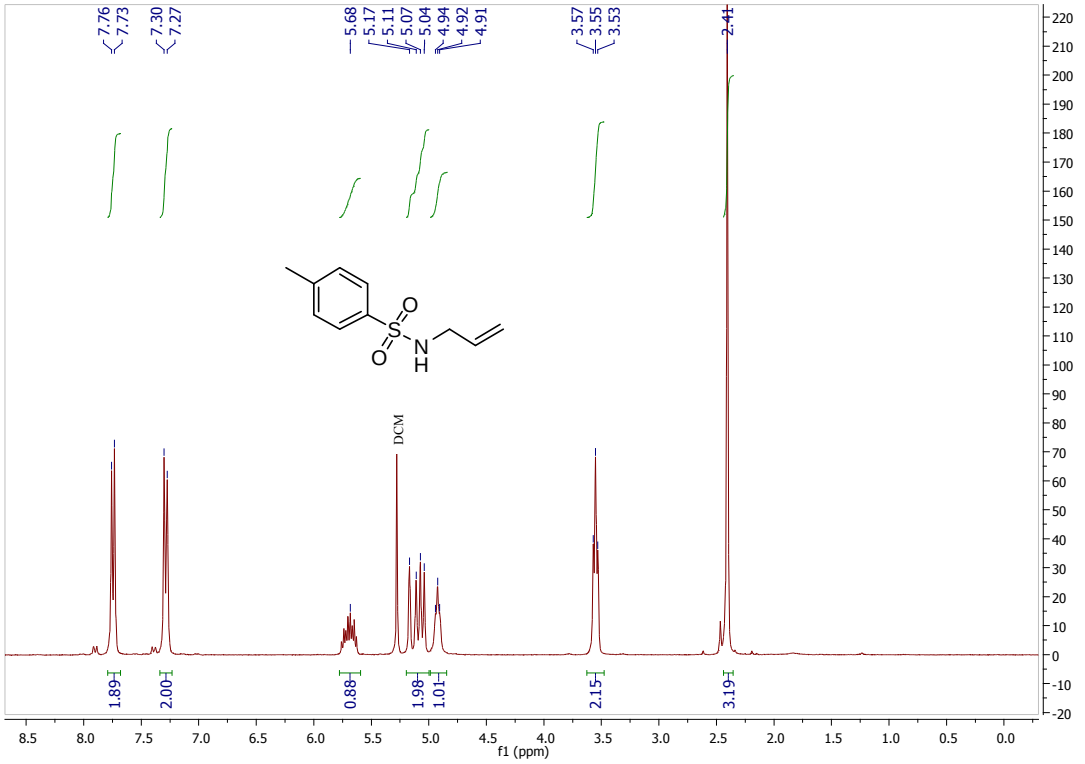
47.10
34.37
27.55
21.73
19.98
f1 (ppm)

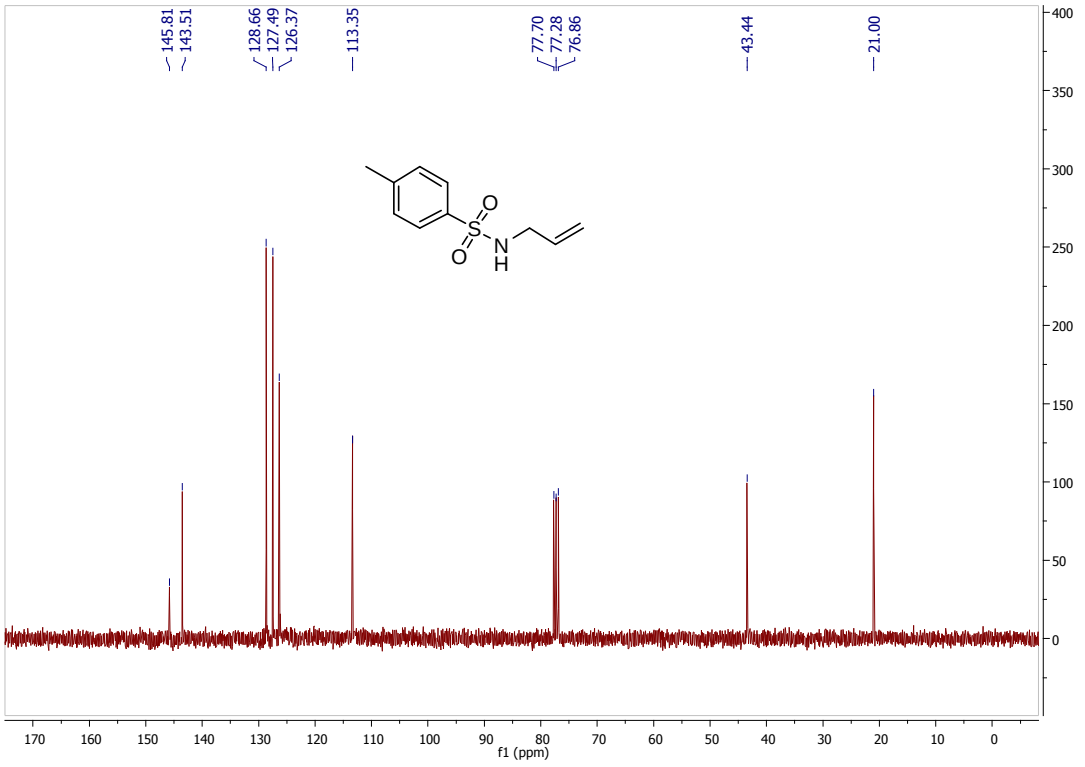
(S)-4-methyl-N-(2-methylbut-3-en-1-yl)-N-(4-methylenehex-5-en-1-yl)benzenesulfonamide (8c)



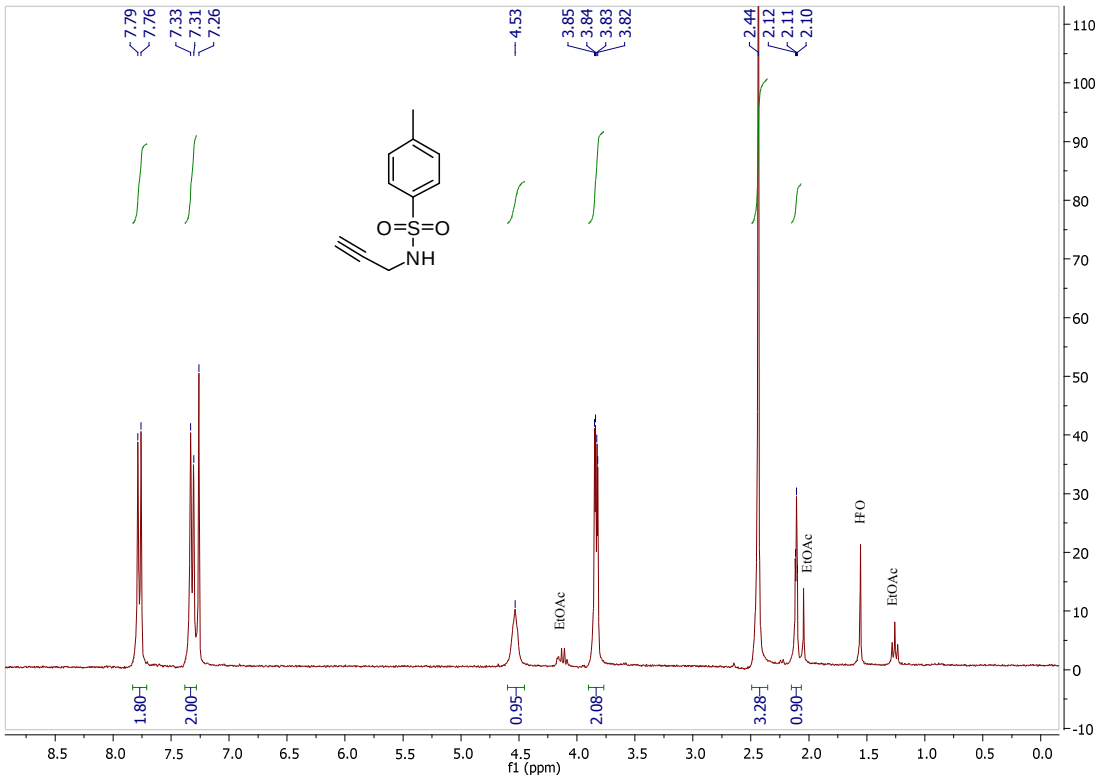


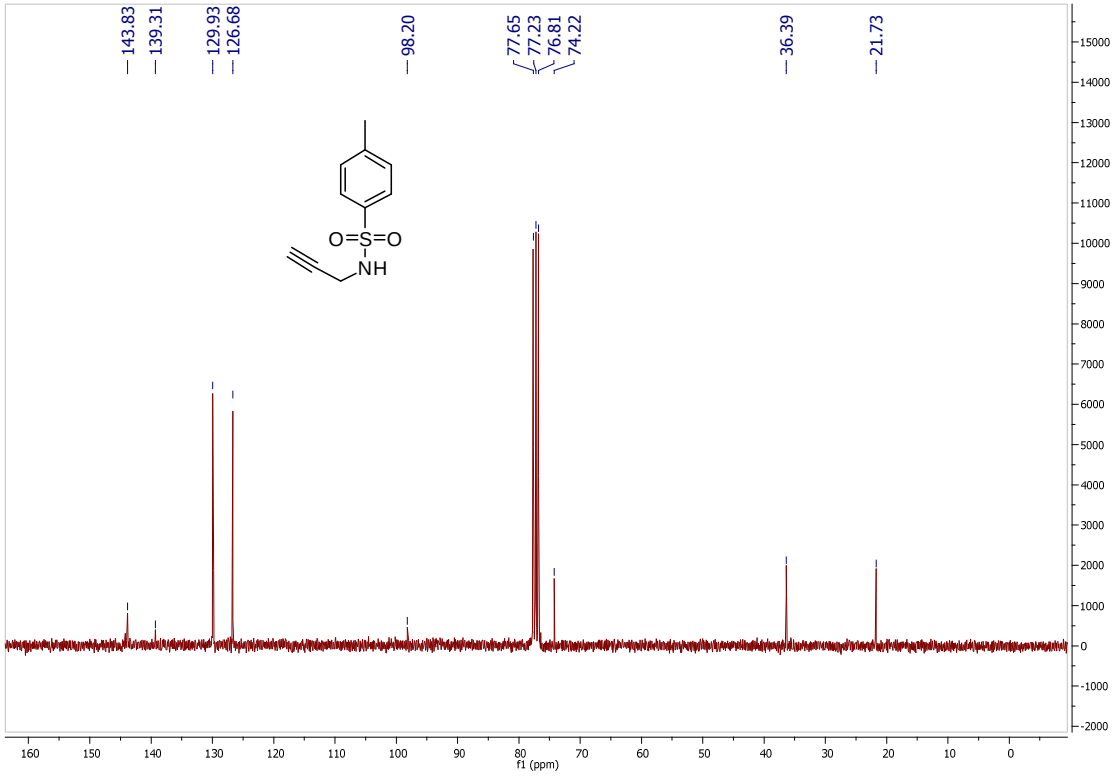
N-allyl-4-methylbenzenesulfonamide (9)



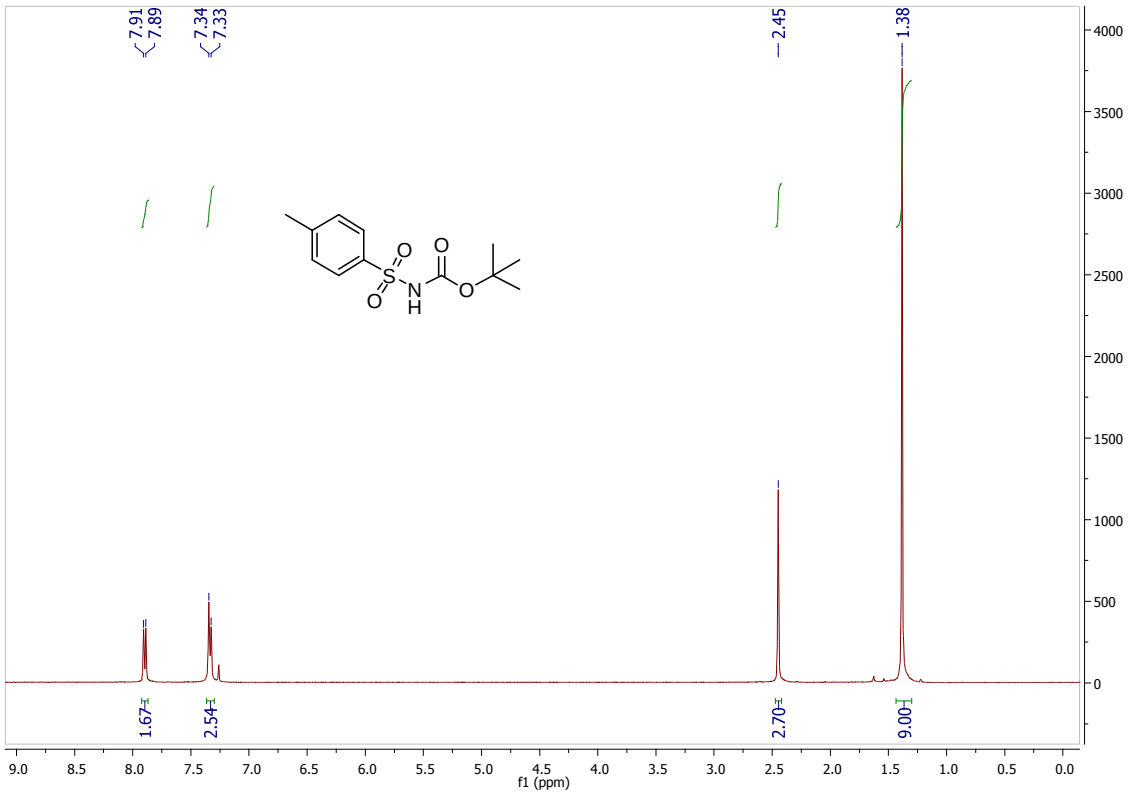


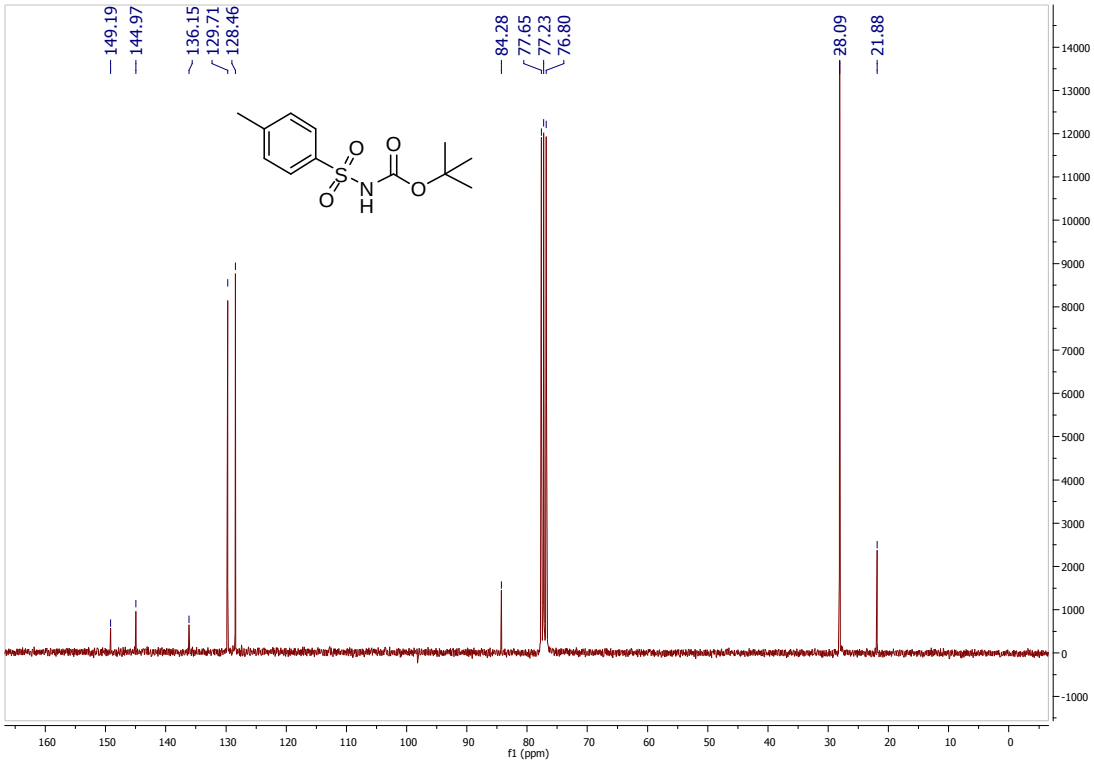
4-Methyl-N-(prop-2-yn-1-yl)benzenesulfonamide (10)



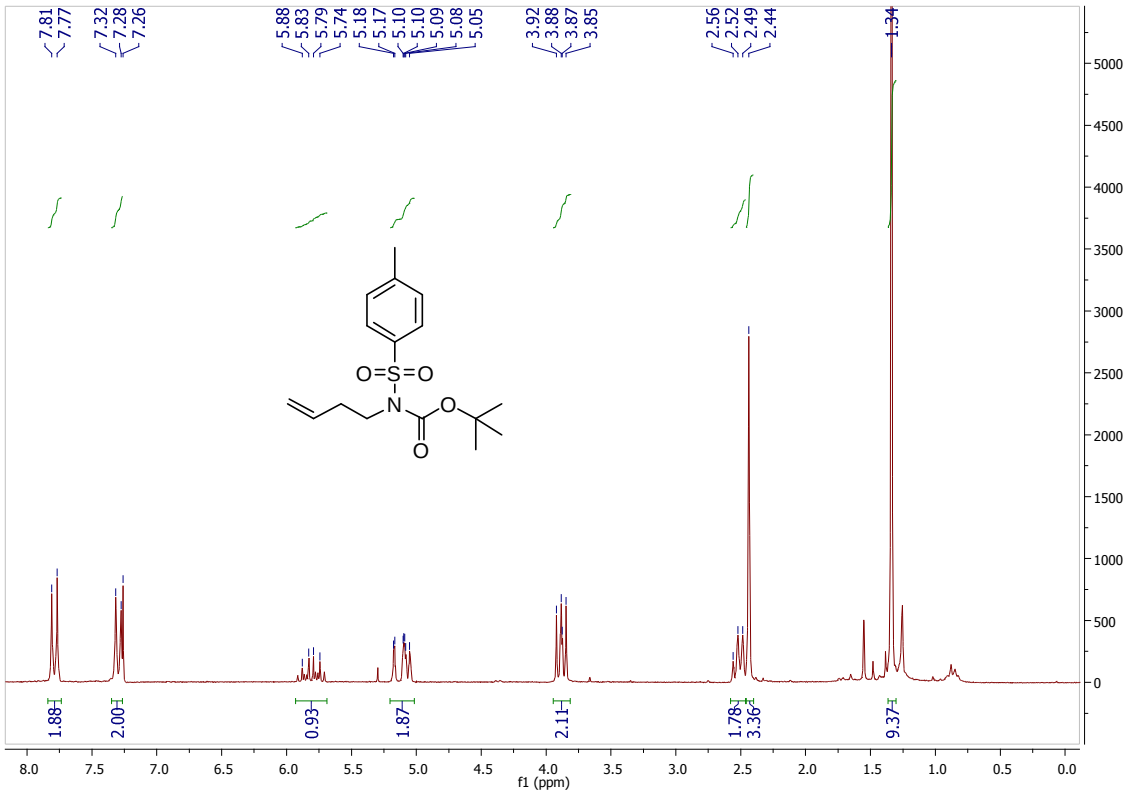


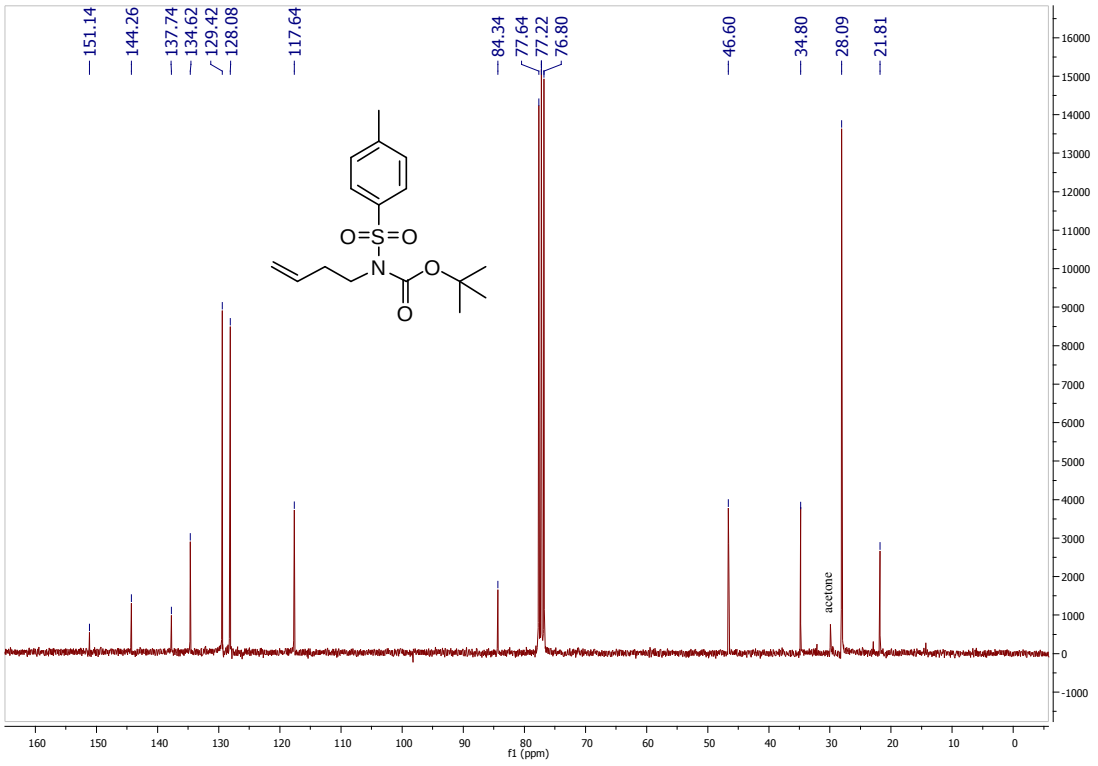
tert-Butyl tosylcarbamate (11)



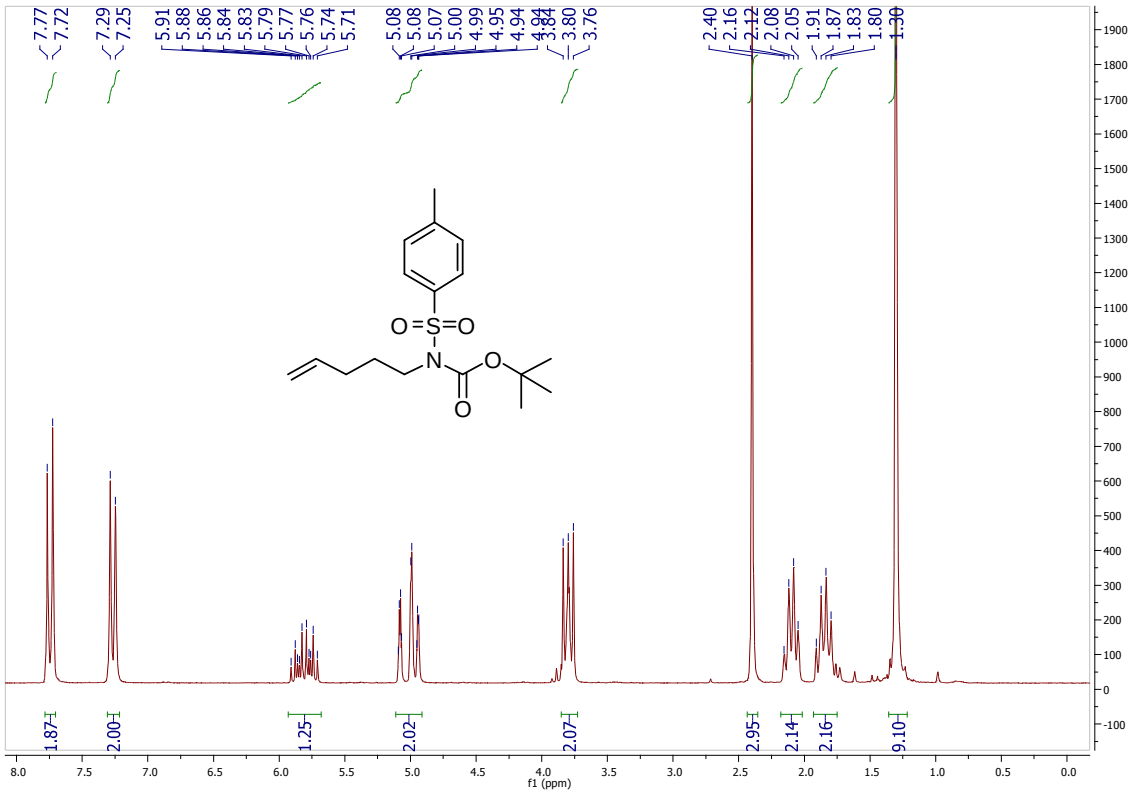


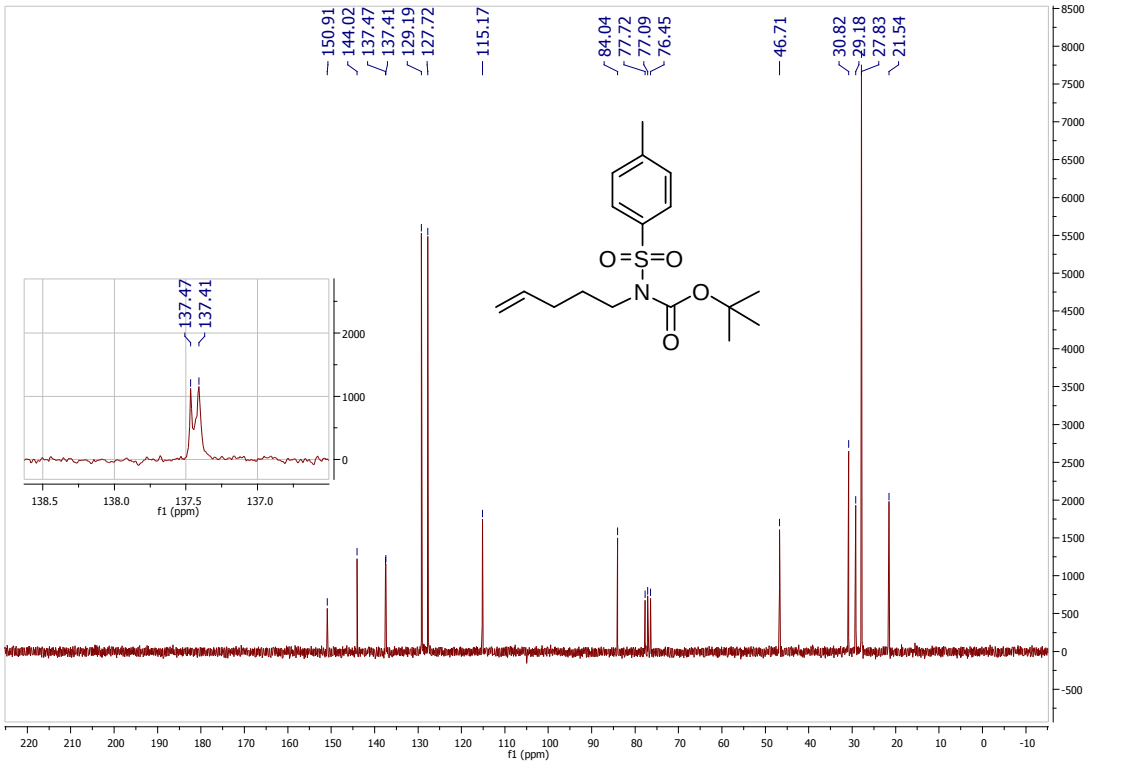
(*N*-*tert*-Butoxycarbonyl)(but-3-enyl)tosylamide (12a)



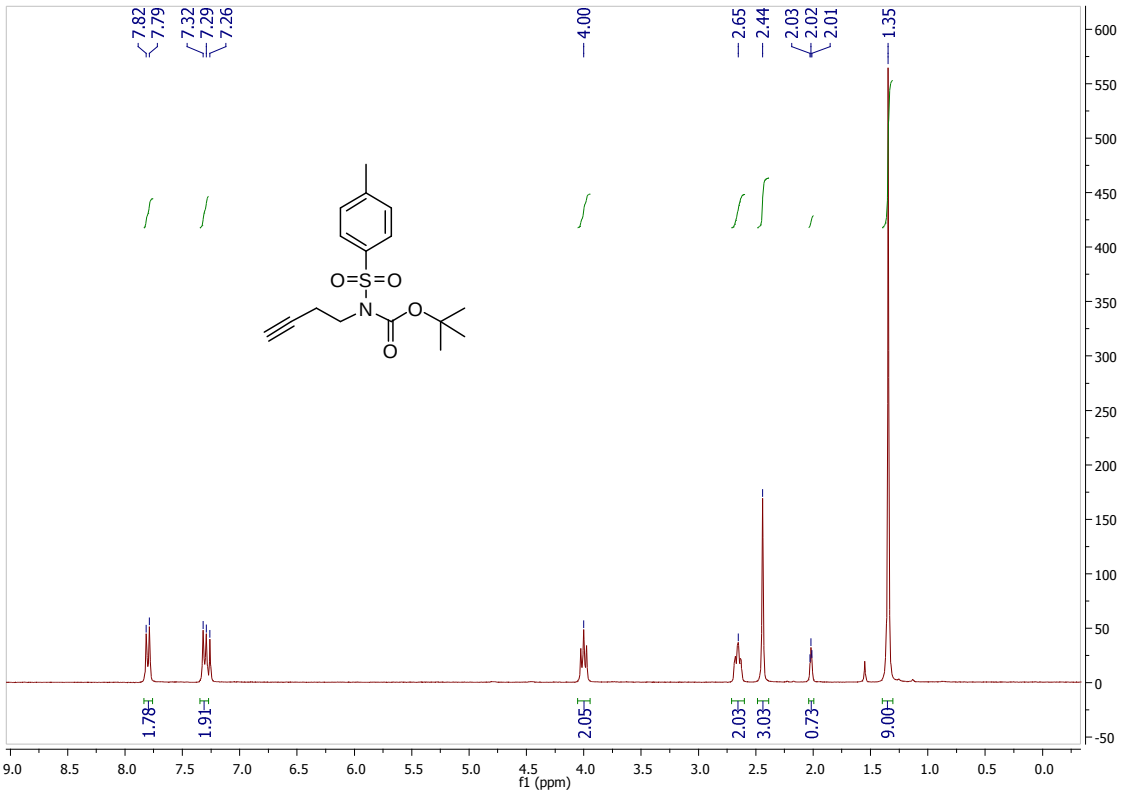


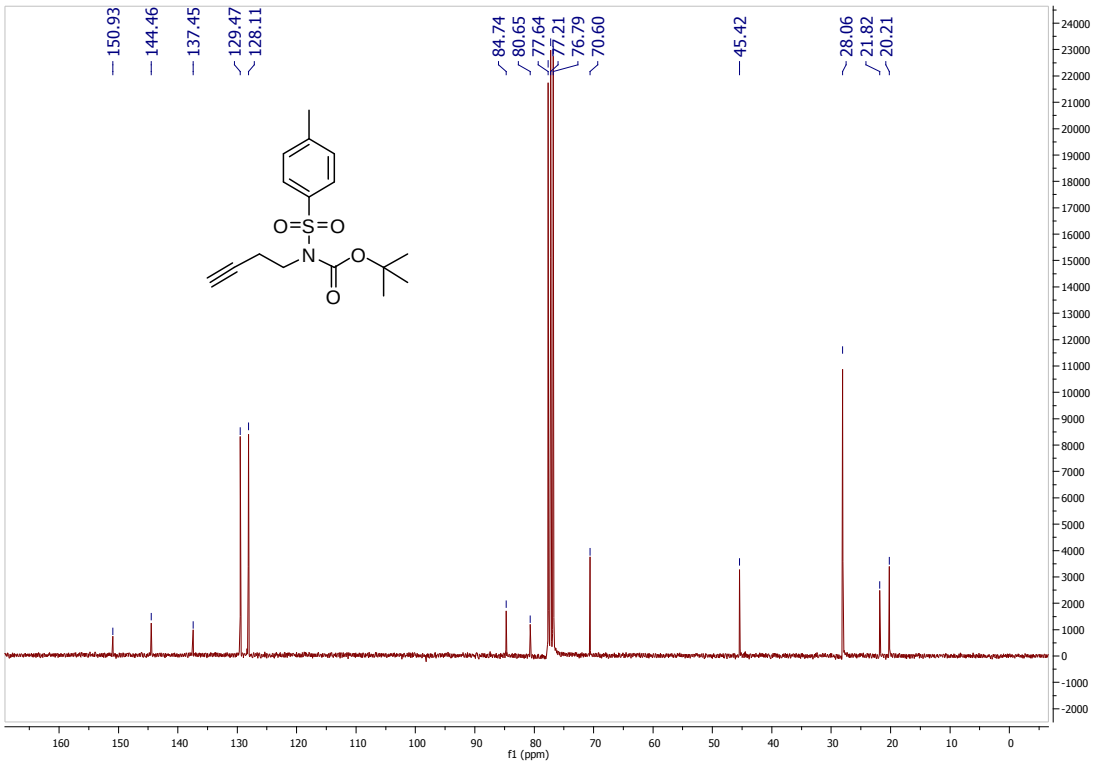
(*N-tert*-Butoxycarbonyl)(but-3-ynyl)tosylamide (12b)



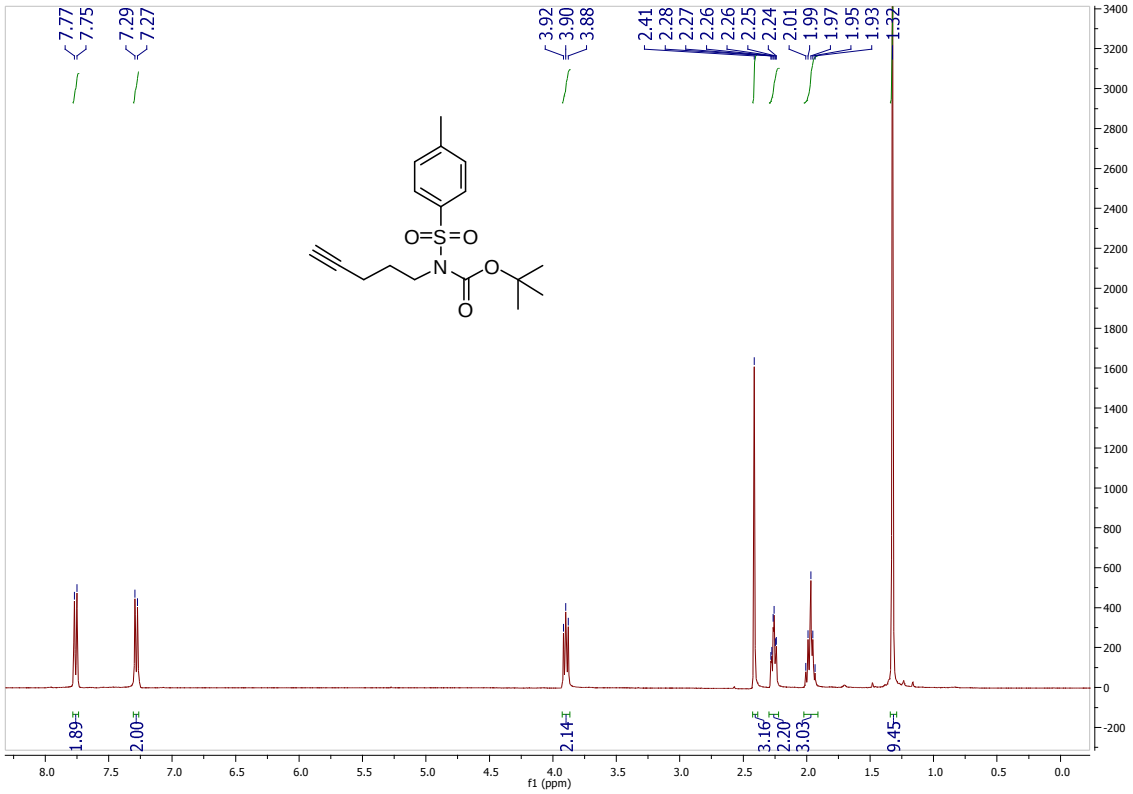


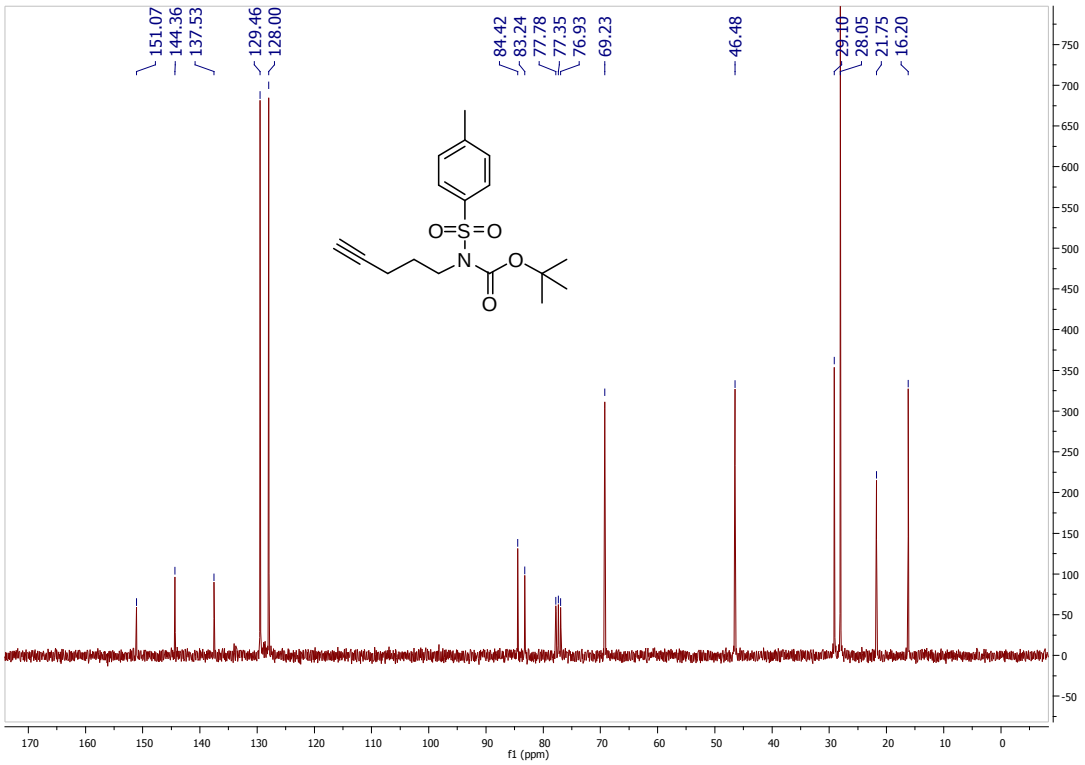
(*N*-*tert*-Butoxycarbonyl)(but-3-ynyl)tosylamide (13a)



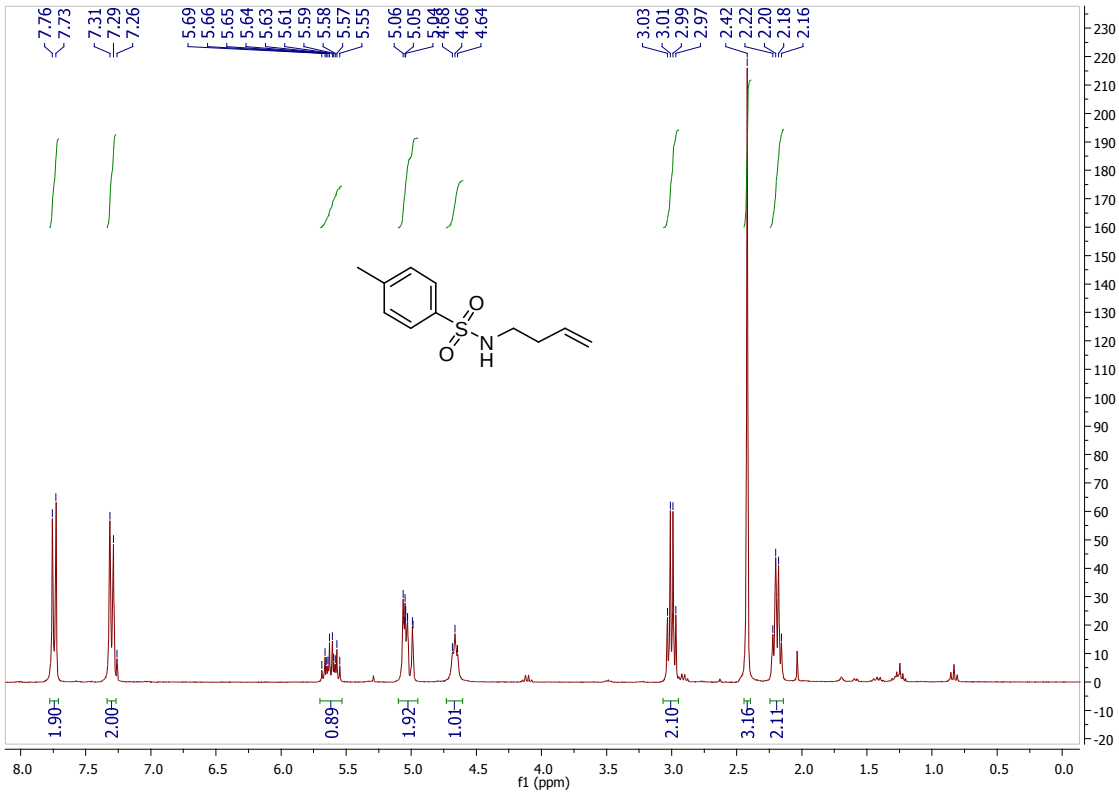


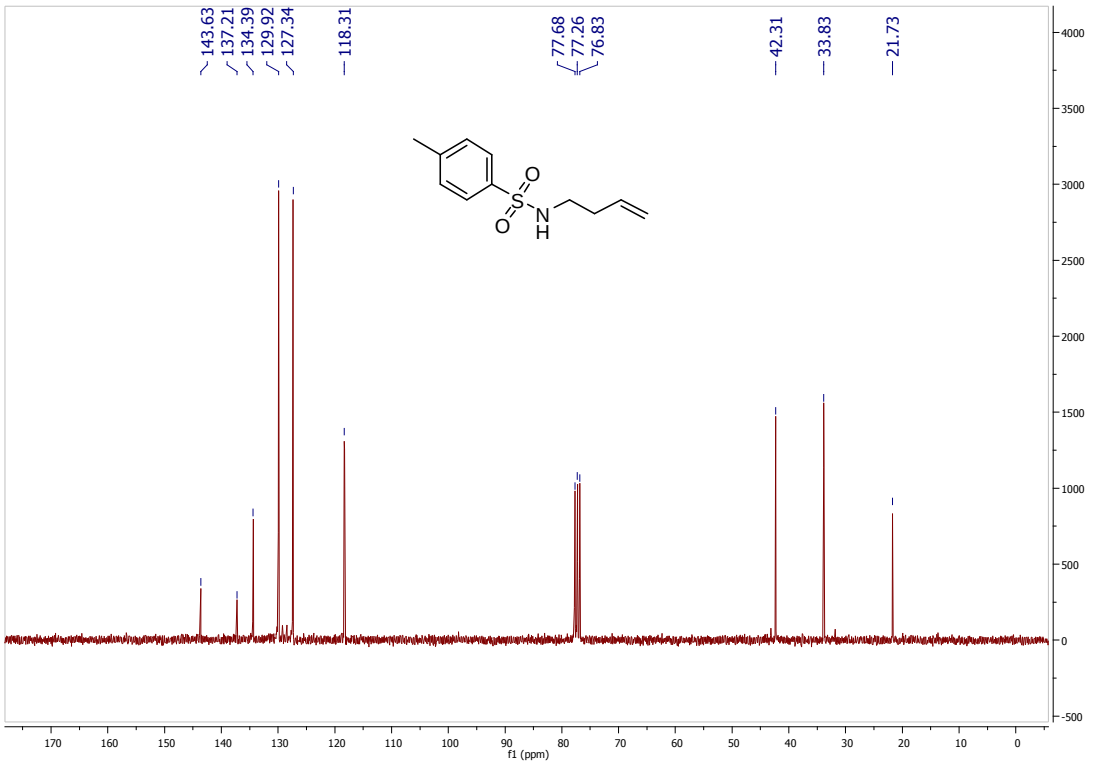
(*N*-*tert*-Butoxycarbonyl)(pent-4-ynyl)tosylamide (13b)



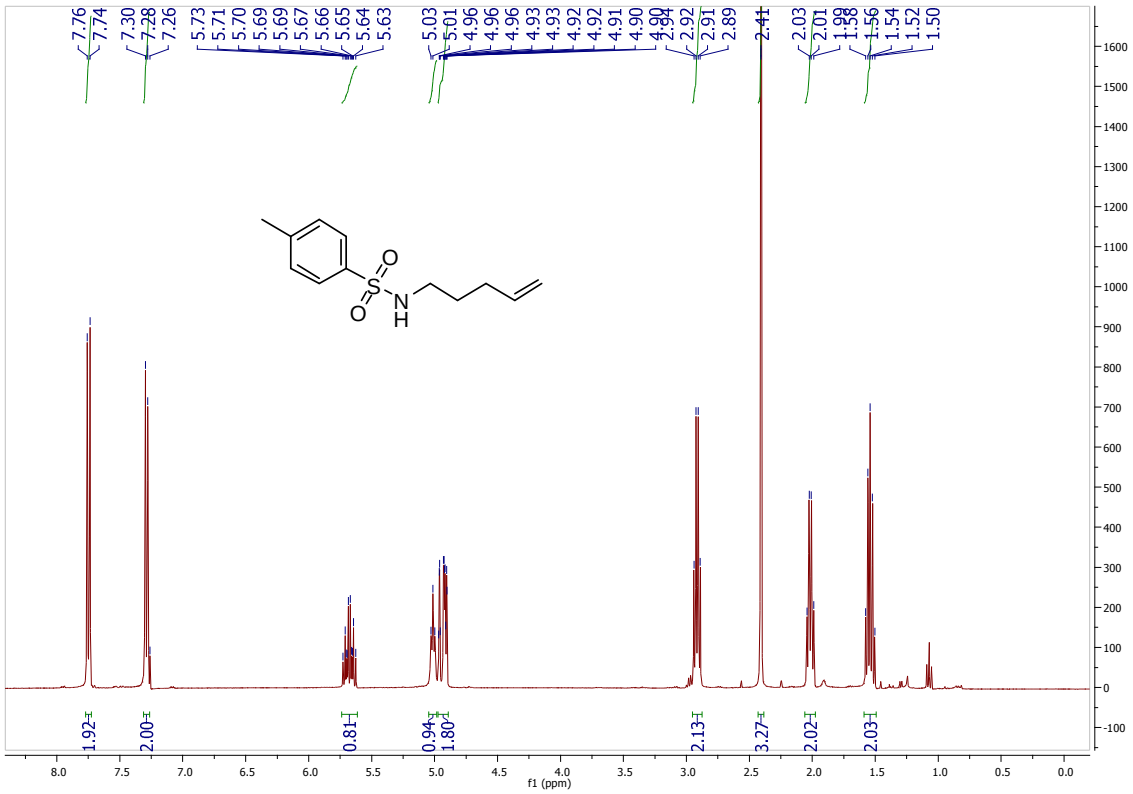


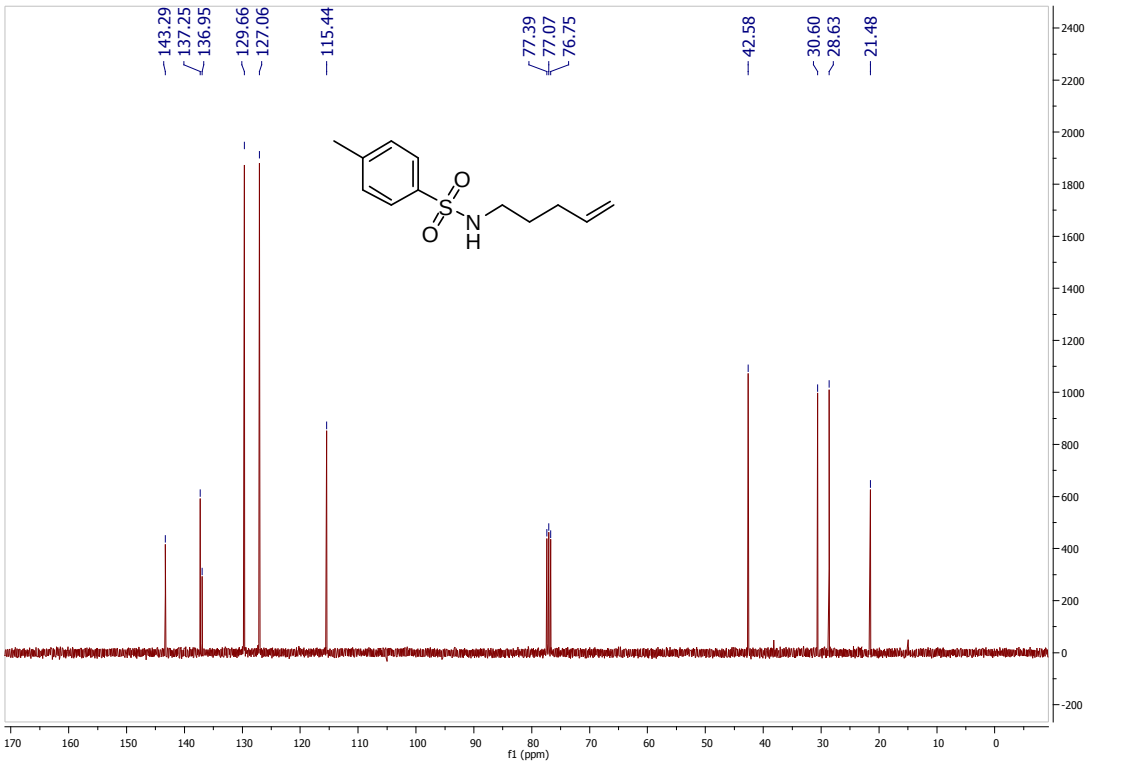
N-3-Buten-1-yl-4-methyl-benzenesulfonamide (14a)



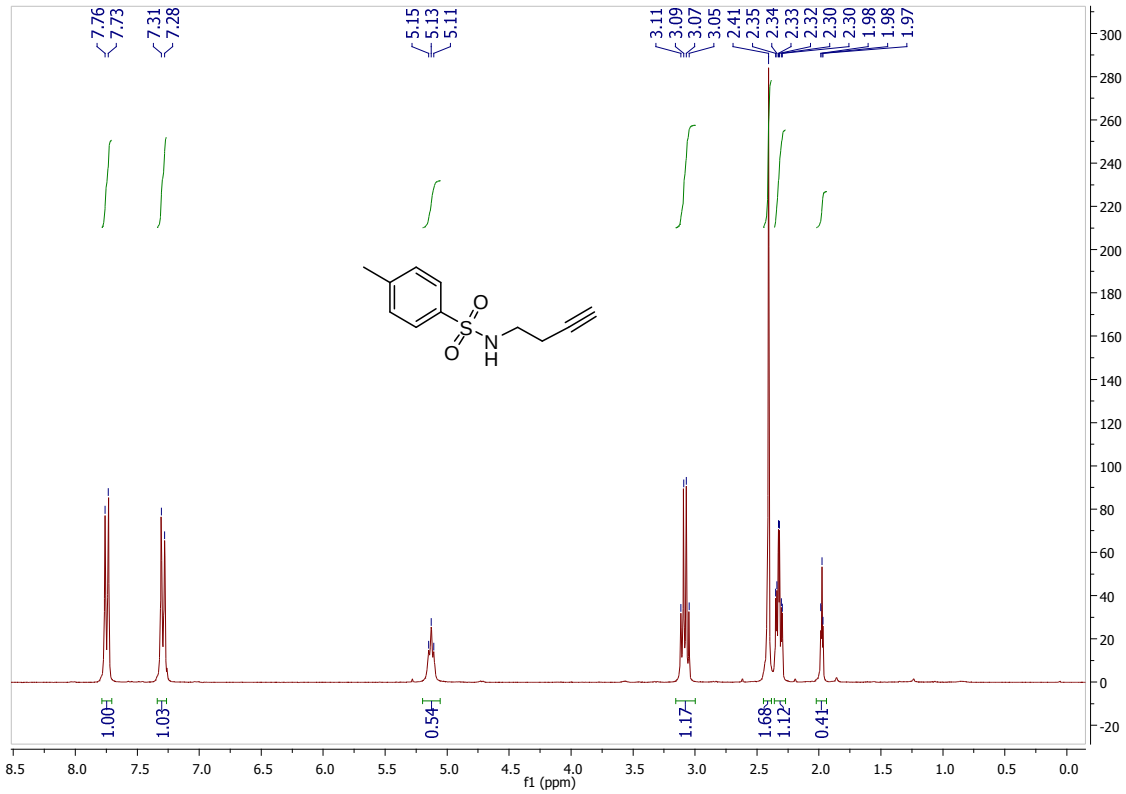


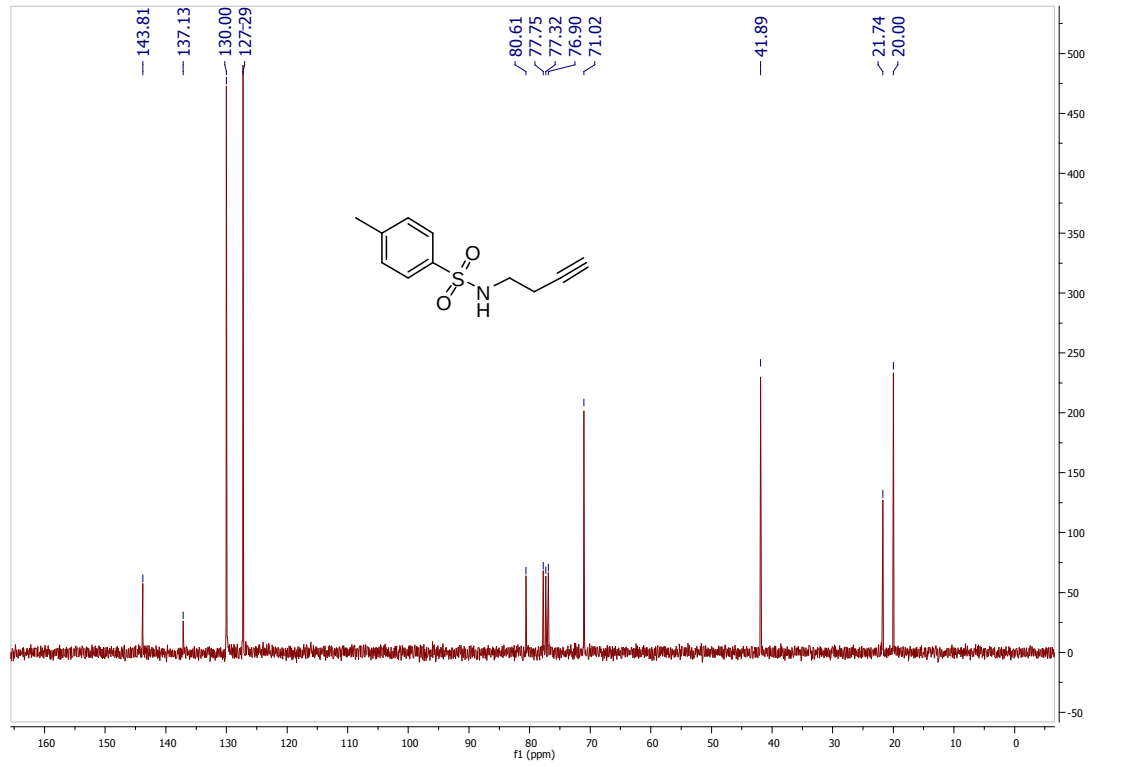
_N_-4-penten-1-yl-4-methyl-benzenesulfonamide (14b)



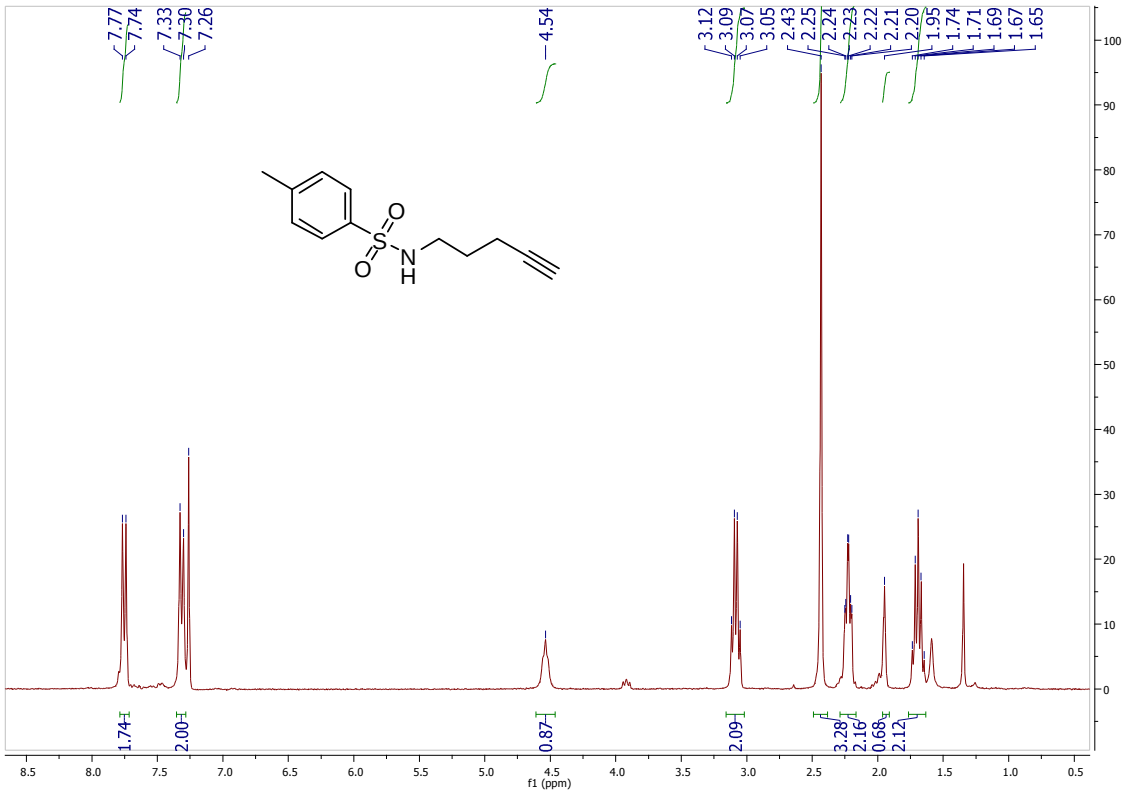


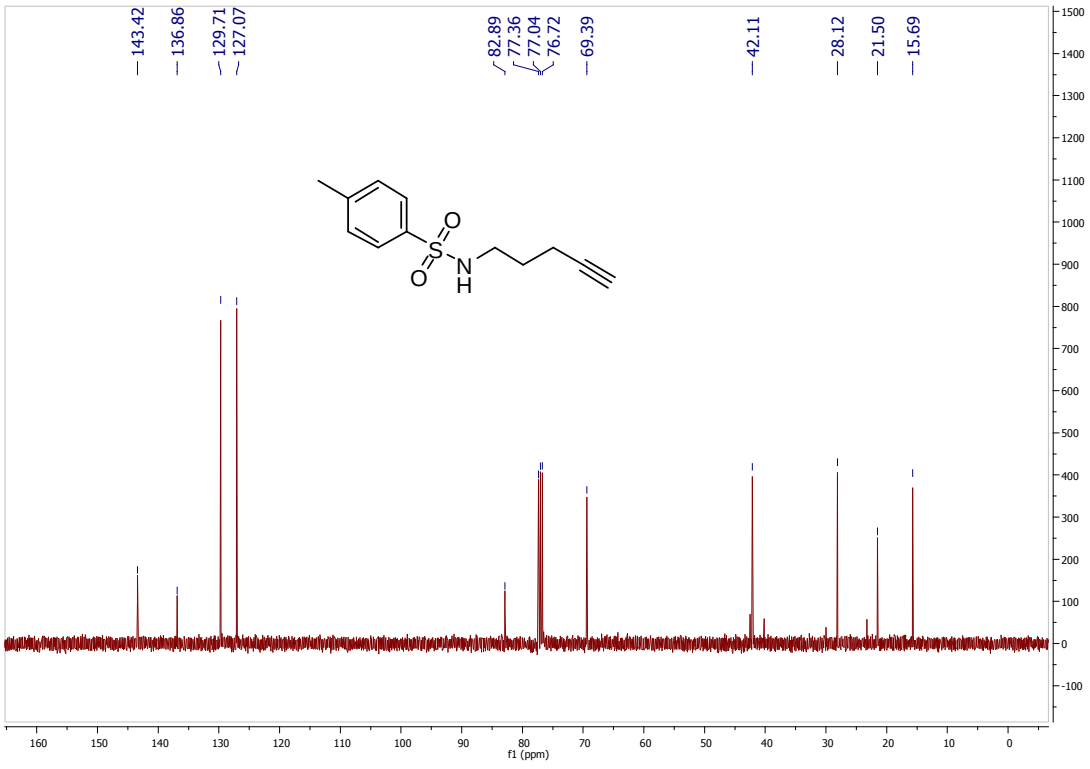
_N_-3-butyn-1-yl-4-methyl-benzenesulfonamide (15a)





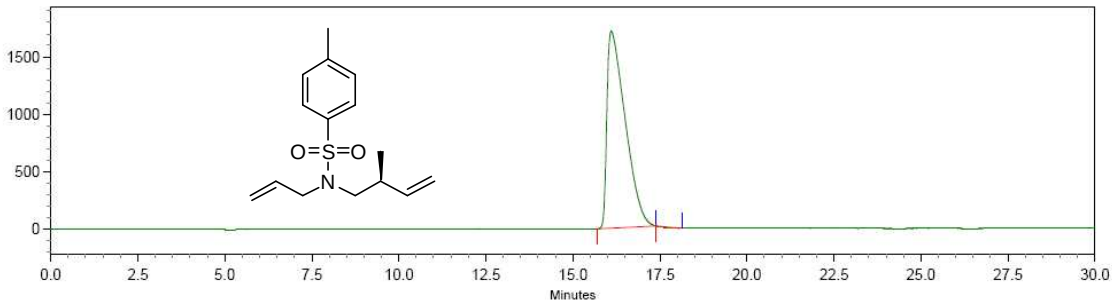
_N_-4-pentyn-1-yl-4-methyl-benzenesulfonamide (15b)


7.77
7.74
7.33
7.30
7.26
4.54
3.12
3.09
3.07
3.05
2.43
2.25
2.24
2.23
2.22
2.21
2.20
1.95
1.74
1.71
1.69
1.67
1.65
1.74
2.00
0.87
2.09
3.28
2.16
0.68
2.12
f1 (ppm)


143.42
136.86
129.71
127.07
82.89
77.36
77.04
76.72
69.39
42.11
28.12
21.50
15.69
f1 (ppm)

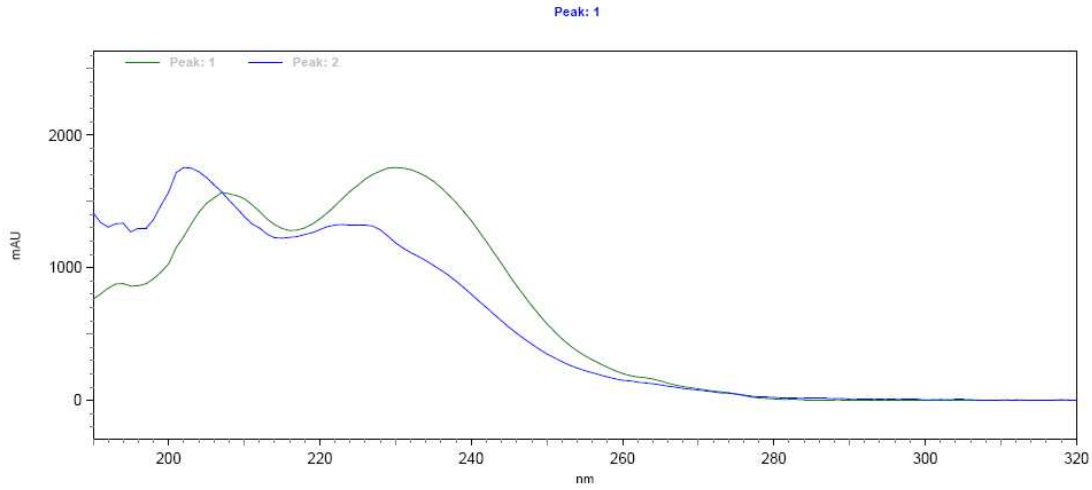
(S)-*N*-Allyl-4-methyl-*N*-(2-methylbut-3-en-1-yl)benzenesulfonamide (2a)

Sample ID: SZA009 **Data Name:** F:\NMR\20091202_30
User: System **Method Name:** C:\CLASS-VP\Methods\Algemeen\Chiralpak AD\AD 99_1 30min.met
Vial # : 3 **Inj. Vol :** 1 ul
Sample Amt: 1
Acquired: 12/4/2009 3:37:24 PM **Printed:** 7/16/2010 5:28:48 PM



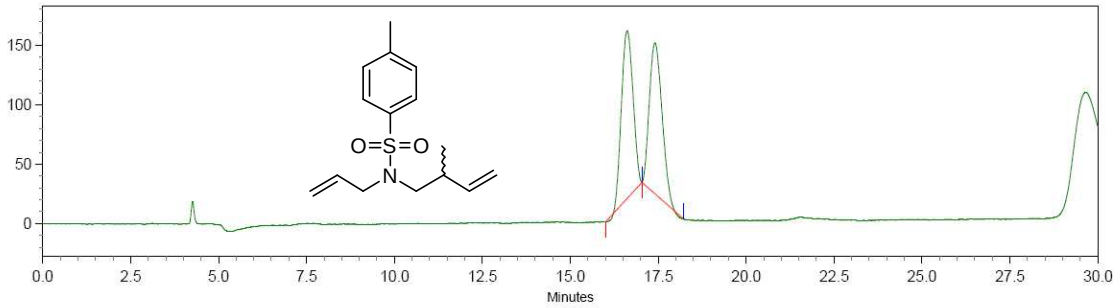
1: 230 nm, 8 nm

Pk #	Name	Retention Time	Area	Area Percent
1	1	16.107	62601461	99.69
2	2	17.397	191734	0.31
Totals			62793195	100.00



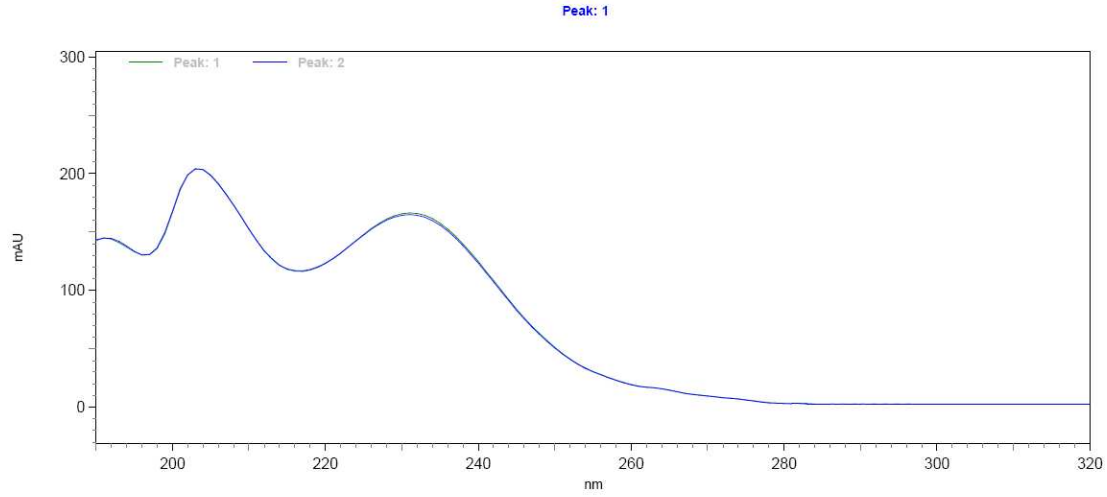
(2a) – Racemic

Sample ID: SZA012 **Data Name:** F:\NMR\20091202_29
User: System **Method Name:** C:\CLASS-VP\Methods\Algemeen\Chiralpak AD\AD 99_1 30min.met
Vial # : 1 **Inj. Vol :** 1 ul
Sample Amt: 1
Acquired: 12/4/2009 2:35:29 PM **Printed:** 7/16/2010 5:27:59 PM



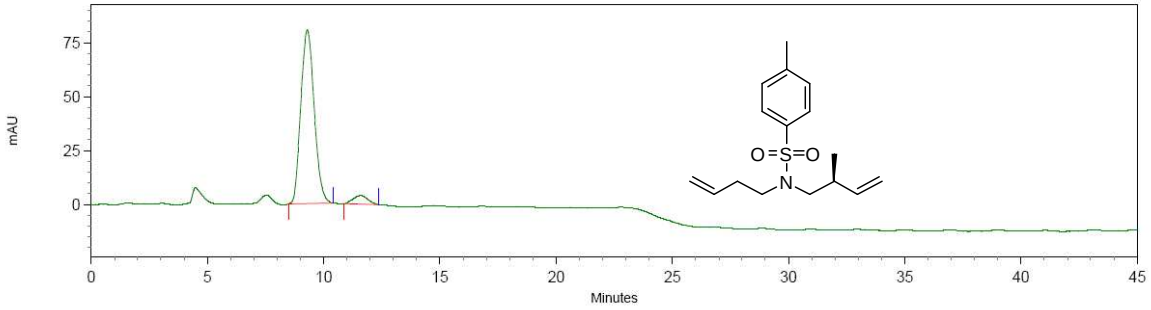
1: 230 nm, 8 nm

Pk #	Name	Retention Time	Area	Area Percent
1	1	16.619	3219988	50.20
2	2	17.408	3194100	49.80
Totals			6414088	100.00



(S)-*N*-(but-3-en-1-yl)-4-methyl-*N*-(2-methylbut-3-en-1-yl)benzenesulfonamide (2b)

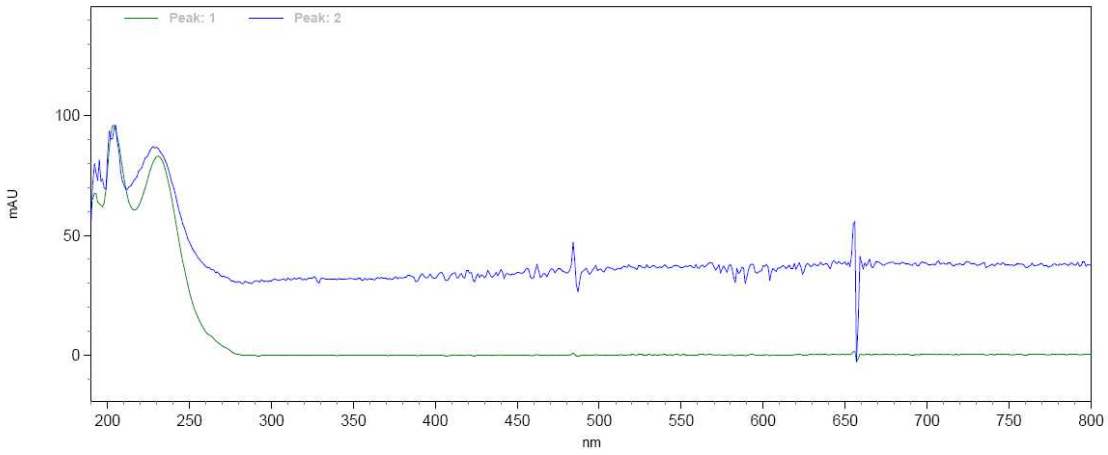
Sample ID: SZA050 **Data Name:** C:\CLASS-VP\Data\20100219\20100219_08
User: System **Method Name:** C:\CLASS-VP\Methods\Algemeen\Chiralcel OJ\OJ 99_1 45min.met
Vial # : 26 **Inj. Vol :** 1 ul
Sample Amt: 1
Acquired: 2/19/2010 7:49:47 PM **Printed:** 7/16/2010 4:41:06 PM



1: 230 nm, 8 nm

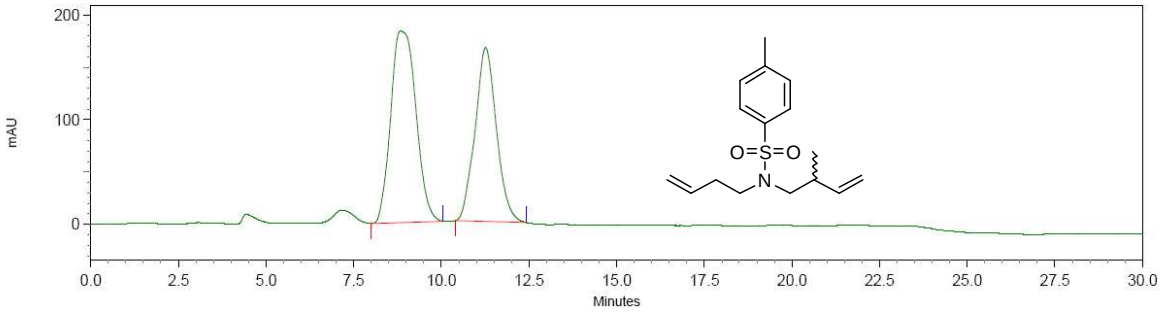
Pk #	Name	Retention Time	Area	Area Percent
1	1	9.301	3229529	94.85
2	2	11.573	175178	5.15
Totals			3404707	100.00

Peak: 1



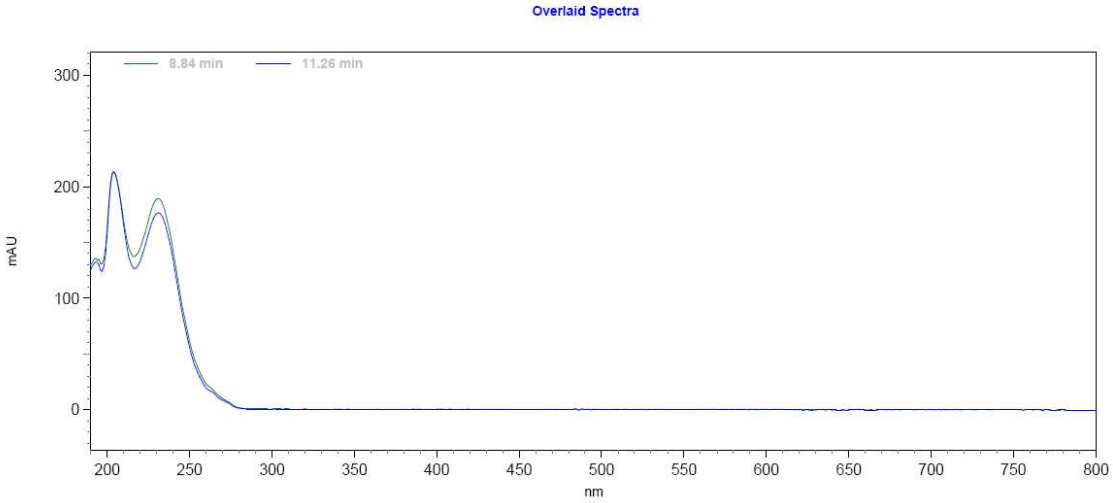
(2b) – Racemic

Sample ID:	**SZA047**	**Data Name:**	**C:\CLASS-VP\Data\20100222\20100222_07**
User:	**System**	**Method Name:**	**C:\CLASS-VP\Methods\Algemeen\Chiralcel OJ\OJ 99_1 30min.met**
Vial # :	**22**	**Inj. Vol :**	**1 ul**
Sample Amt:	**1**		
Acquired:	**2/22/2010 8:15:38 PM**	**Printed:**	**7/16/2010 4:30:44 PM**



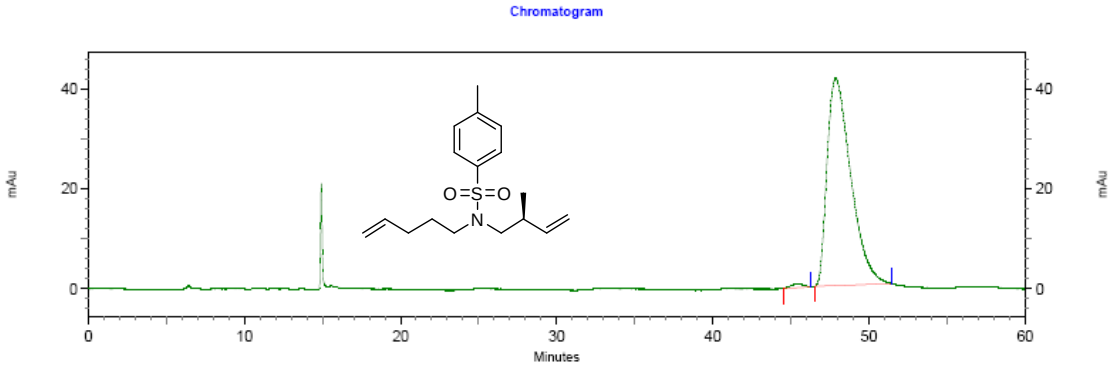
1: 230 nm, 8 nm

Pk #	Name	Retention Time	Area	Area Percent
1	1	8.843	9170528	56.51
2	2	11.264	7056226	43.49
Totals			16226754	100.00



(S)-4-methyl-*N*-(2-methylbut-3-en-1-yl)-*N*-(pent-4-en-1-yl)benzenesulfonamide (2c)

```
Sample ID      :    SZA091
Vial#          :    89
Sample amount  :    1
Inj. volume    :    1
Acquired       :    14-7-2010 11:04:39
Data Name      :    D:\DATA\20100713\20100713_13
Method Name    :
C:\CLASS-VP\Enterprise\Projects\Default\Method\General\Chiralpak OD-H\OD-H
995_5 60 min.met
Sequence Name  :
C:\CLASS-VP\Enterprise\Projects\Default\Sequence\20100712.seq
```



1: 230 nm,
2 nm Results

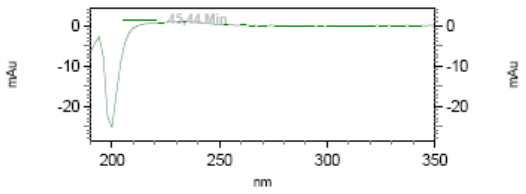
Pk #	Name	Retention Time	Area	Area Percent
1	1	45,436	41361	0,933
2	2	47,876	4391466	99,067
Totals			4432827	100,000

Spectrum Report

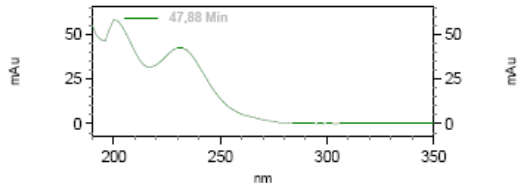
Spectra of all named detected peaks

(The peak spectrum is defined as the peak apex spectrum)

Multi-Chrom 1 (1: 230 nm, 2 nm)
Spectra



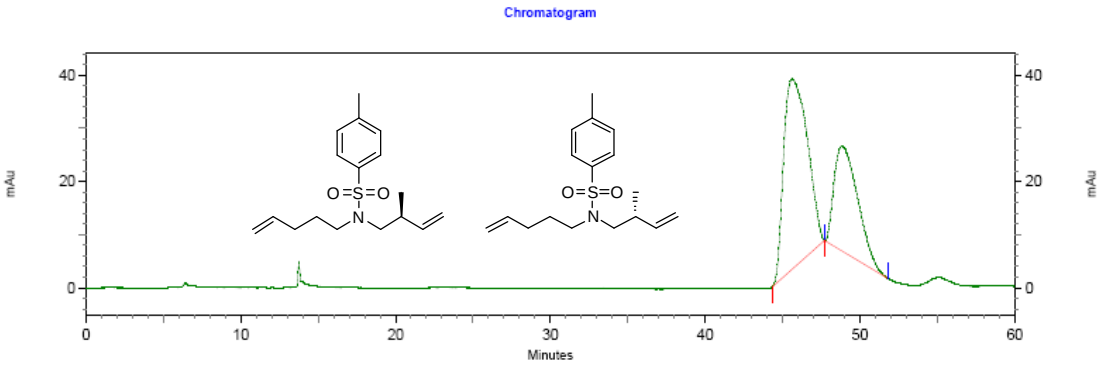
Retention time: 45,436 Min
Peak name: 1
Lambda max: 231, 338, 326
Lambda min: 200, 299, 334



Retention time: 47,876 Min
Peak name: 2
Lambda max: 200, 231, 325
Lambda min: 335, 339, 301

(2c) – Racemic mixture

Sample ID : SZA090+91
Vial# : 90
Sample amount : 1
Inj. volume : 1
Acquired : 14-7-2010 11:12:59
Data Name : D:\DATA\20100713\20100713_12
Method Name :
C:\CLASS-VP\Enterprise\Projects\Default\Method\General\Chiralpak OD-H\OD-H 995_5 60 min.met
Sequence Name :
C:\CLASS-VP\Enterprise\Projects\Default\Sequence\20100712.seq



1: 230 nm,
2 nm Results

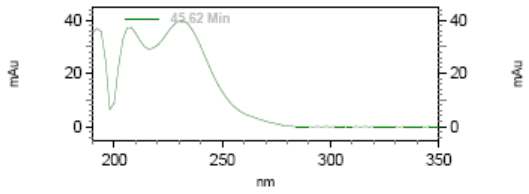
Pk #	Name	Retention Time	Area	Area Percent
1	1	45,624	3784570	63,955
2	2	48,848	2132989	36,045
Totals			5917559	100,000

Spectrum Report

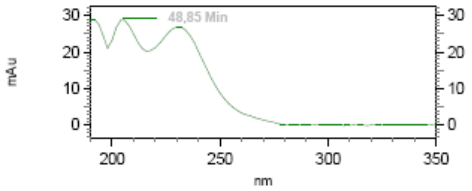
Spectra of all named detected peaks

(The peak spectrum is defined as the peak apex spectrum)

Multi-Chrom 1 (1: 230 nm, 2 nm)
Spectra



Retention time: 45,624 Min
Peak name: 1
Lambda max: 231, 210, 323
Lambda min: 303, 338, 298

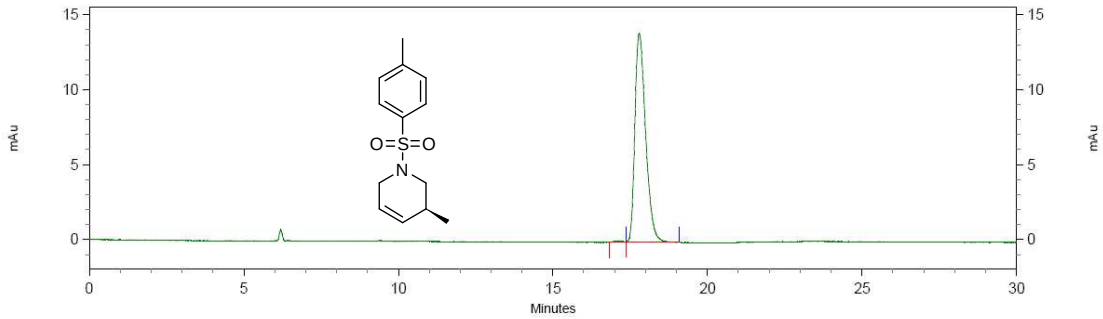


Retention time: 48,848 Min
Peak name: 2
Lambda max: 205, 230, 323
Lambda min: 304, 325, 338

(S)-3-Methyl-1-tosyl-1,2,3,6-tetrahydropyridine (4a)

Sample ID : SZA011
Vial# : 20
Sample amount : 1
Inj. volume : 1
Acquired : 1-12-2009 13:48:20
Data Name : E:\20091130\20091127_11
Method Name :
C:\CLASS-VP\Enterprise\Projects\Default\Method\General\Chiralpak AS-H\AS-H 95_5 30 min.met
Sequence Name : C:\CLASS-VP\Enterprise\Projects\Default\Sequence\All 2009\20091130.seq

Chromatogram



1: 254
nm, 4 nm
Results

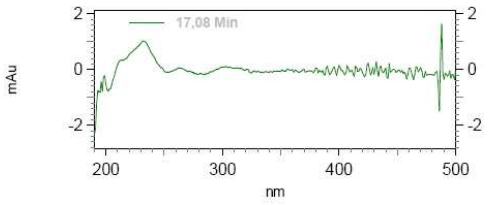
Pk #	Name	Retention Time	Area	Area Percent
1	Peak @ 17,084 Minutes	17,084	2125	0,624
2	Peak @ 17,792 Minutes	17,792	338304	99,376
Totals			340429	100,000

Spectrum Report

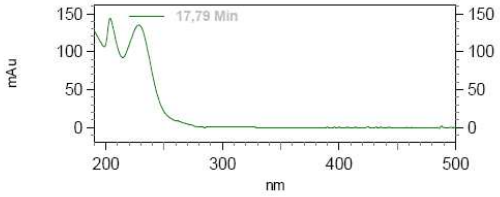
Spectra of all named detected peaks

(The peak spectrum is defined as the peak apex spectrum)

Multi-Chrom 1 (1: 254 nm, 4 nm)
Spectra



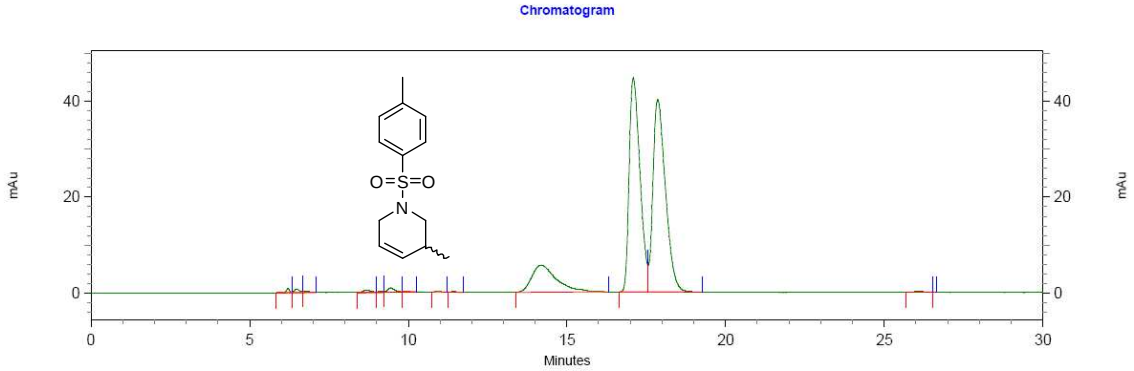
Retention time: 17,084 Min
Peak name: Peak @ 17,084 Minutes
Lambda max: 232, 303, 459
Lambda min: 203, 441, 493



Retention time: 17,792 Min
Peak name: Peak @ 17,792 Minutes
Lambda max: 204, 228, 304
Lambda min: 441, 493, 496

(4a) – Racemic

Sample ID : SZA015
Vial# : 21
Sample amount : 1
Inj. volume : 1
Acquired : 10-12-2009 11:07:49
Data Name : E:\20091207\20091207_11
Method Name :
C:\CLASS-VP\Enterprise\Projects\Default\Method\General\Chiralpak AS-H\AS-H 90_10 45 min.met
Sequence Name : C:\CLASS-VP\Enterprise\Projects\Default\Sequence\All 2009\20091130.seq



1: 254
nm, 4 nm
Results

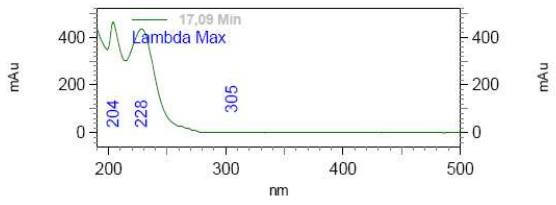
Pk #	Name	Retention Time	Area	Area Percent
10	Peak @ 14,192 Minutes	14,192	307181	12,168
11	Peak @ 17,092 Minutes	17,092	1058294	41,922
12	Peak @ 17,860 Minutes	17,860	1105222	43,781
Totals			2470697	97,871

Spectrum Report

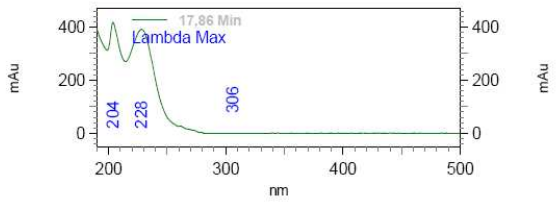
Spectra of all named detected peaks

(The peak spectrum is defined as the peak apex spectrum)

Multi-Chrom 1 (1: 254 nm, 4 nm)
Spectra



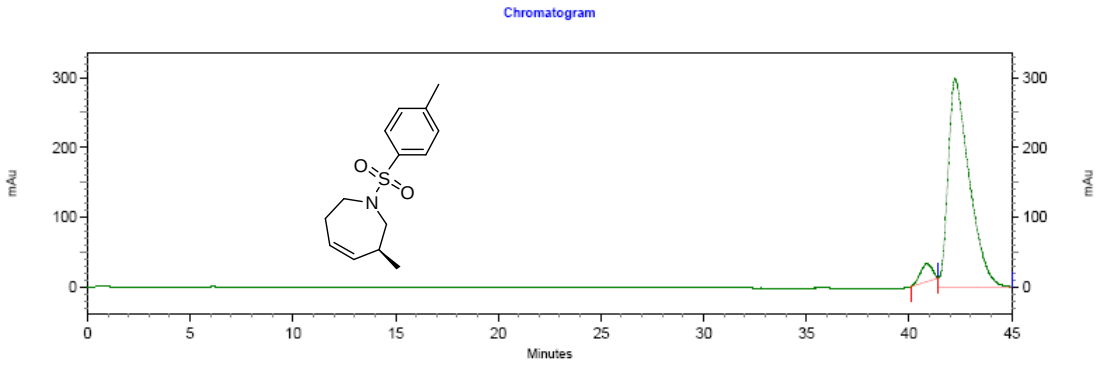
Retention time: 17,092 Min
Peak name: Peak @ 17,092 Minutes
Lambda max: 204, 228, 305
Lambda min: 441, 391, 493



Retention time: 17,860 Min
Peak name: Peak @ 17,860 Minutes
Lambda max: 204, 228, 306
Lambda min: 441, 493, 497

(S)-3-methyl-1-tosyl-2,3,6,7-tetrahydro-1H-azepine (4b)

Sample ID : SZA048
Vial# : 21
Sample amount : 1
Inj. volume : 2
Acquired : 5-3-2010 11:22:58
Data Name : D:\DATA\20100303\20100303_08
Method Name :
C:\CLASS-VP\Enterprise\Projects\Default\Method\General\Chiralpak OJ-H\OJ-H 99_1 45 min.met
Sequence Name :
C:\CLASS-VP\Enterprise\Projects\Default\Sequence\20100712.seq



1: 240
nm, 2 nm
Results

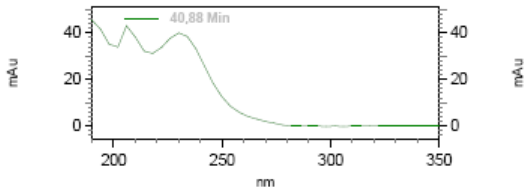
Pk #	Name	Retention Time	Area	Area Percent
1	1	40,880	1022818	4,555
2	2	42,244	21432323	95,445
Totals			22455141	100,000

Spectrum Report

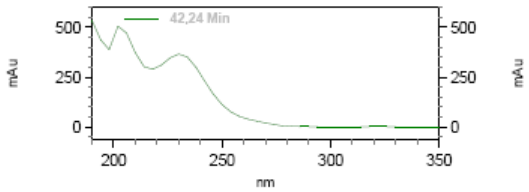
Spectra of all named detected peaks

(The peak spectrum is defined as the peak apex spectrum)

Multi-Chrom 1 (1: 230 nm, 4 nm)
Spectra



Retention time: 40,880 Min
Peak name: 1
Lambda max: 219, 326
Lambda min: 301, 337, 214

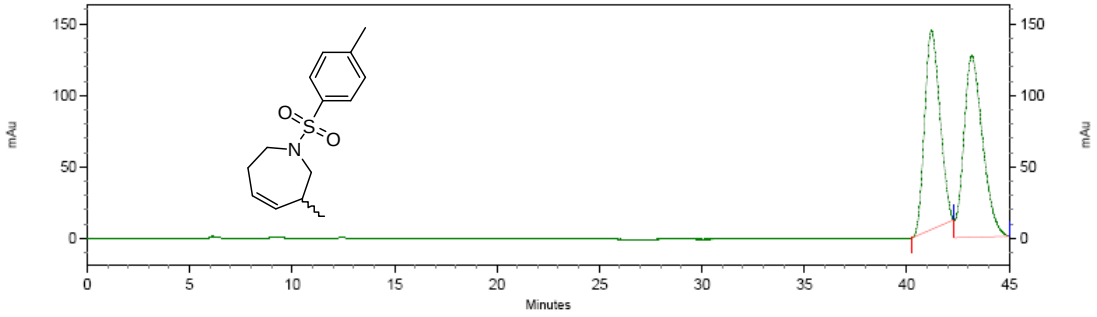


Retention time: 42,244 Min
Peak name: 2
Lambda max: 314
Lambda min: 306

(4b) – Racemic

Sample ID : SZA054
Vial# : 20
Sample amount : 1
Inj. volume : 2
Acquired : 5-3-2010 11:21:03
Data Name : D:\DATA\20100303\20100303_07
Method Name :
C:\CLASS-VP\Enterprise\Projects\Default\Method\General\Chiralpak OJ-H\OJ-H 99_1 45 min.met
Sequence Name :
C:\CLASS-VP\Enterprise\Projects\Default\Sequence\20100712.seq

Chromatogram



1: 240
nm, 2 nm
Results

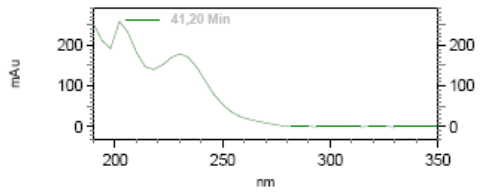
Pk #	Name	Retention Time	Area	Area Percent
1	1	41,204	7205254	47,079
2	2	43,172	8099463	52,921
Totals			15304717	100,000

Spectrum Report

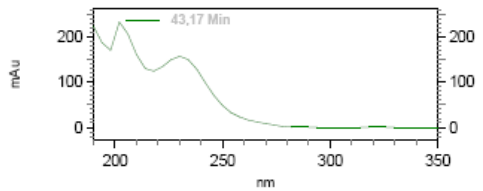
Spectra of all named detected peaks

(The peak spectrum is defined as the peak apex spectrum)

Multi-Chrom 1 (1: 230 nm, 4 nm)
Spectra



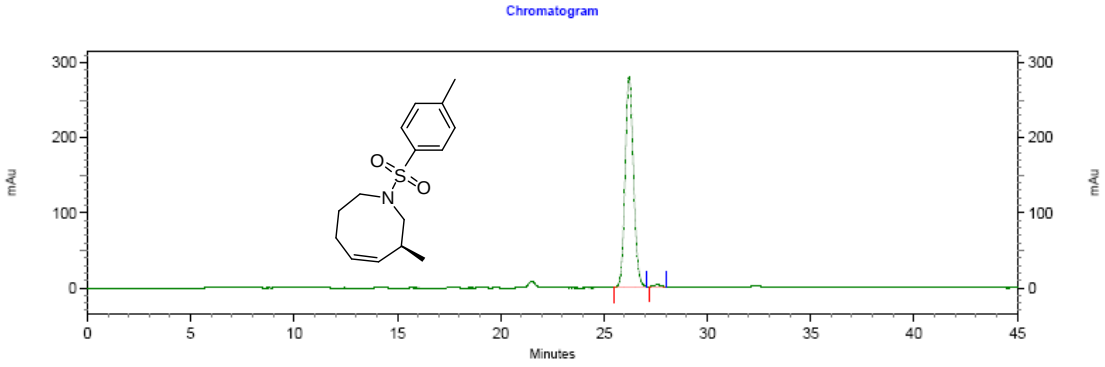
Retention time: 41,204 Min
Peak name: 1
Lambda max: 321, 349
Lambda min: 302, 343



Retention time: 43,172 Min
Peak name: 2
Lambda max: 321, 349
Lambda min: 302, 344

(S)-7-methyl-1-tosyl-1,2,3,4,7,8-hexahydroazocine (4c)

Sample ID : SZA093
Vial# : 58
Sample amount : 1
Inj. volume : 1
Acquired : 14-7-2010 10:41:16
Data Name : D:\DATA\20100713\20100713_07
Method Name :
C:\CLASS-VP\Enterprise\Projects\Default\Method\General\Chiralpak OD-H\OD-H 98_2 45 min.met
Sequence Name :
C:\CLASS-VP\Enterprise\Projects\Default\Sequence\20100712.seq



1: 230 nm,
2 nm Results

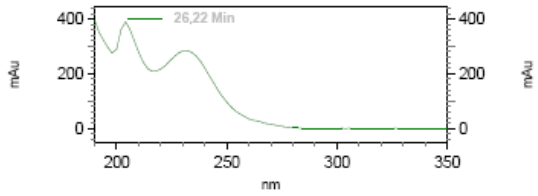
Pk #	Name	Retention Time	Area	Area Percent
1	1	26,216	7911913	99,047
2	2	27,592	76129	0,953
Totals			7988042	100,000

Spectrum Report

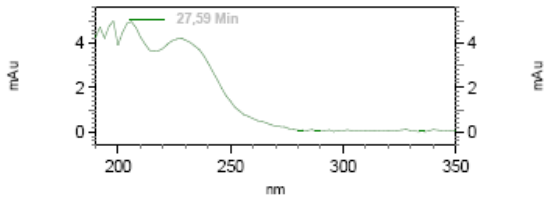
Spectra of all named detected peaks

(The peak spectrum is defined as the peak apex spectrum)

Multi-Chrom 1 (1: 230 nm, 2 nm)
Spectra



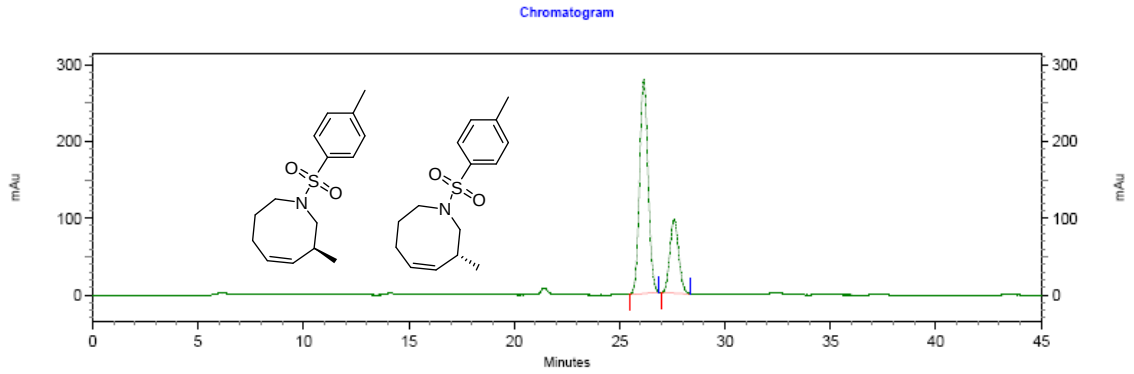
Retention time: 26,216 Min
Peak name: 1
Lambda max: 203, 231, 319
Lambda min: 339, 305, 218



Retention time: 27,592 Min
Peak name: 2
Lambda max: 202, 228, 325
Lambda min: 295, 336, 303

Racemic mixture (**4c**)

Sample ID : SZA093+94
Vial# : 60
Sample amount : 1
Inj. volume : 1
Acquired : 14-7-2010 10:35:48
Data Name : D:\DATA\20100713\20100713_06
Method Name :
C:\CLASS-VP\Enterprise\Projects\Default\Method\General\Chiralpak OD-H\OD-H 98_2 45 min.met
Sequence Name :
C:\CLASS-VP\Enterprise\Projects\Default\Sequence\20100712.seq



1: 230 nm,
2 nm Results

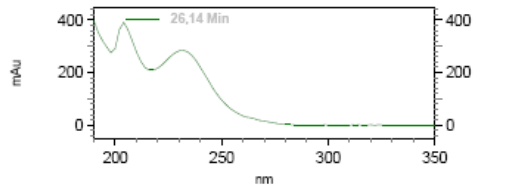
Pk #	Name	Retention Time	Area	Area Percent
1	1	26,144	7886249	73,640
2	2	27,584	2822974	26,360
Totals			10709223	100,000

Spectrum Report

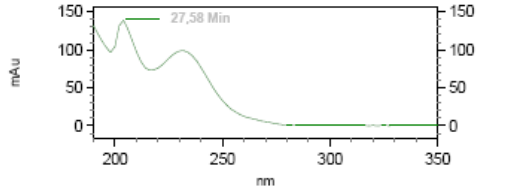
Spectra of all named detected peaks

(The peak spectrum is defined as the peak apex spectrum)

Multi-Chrom 1 (1: 230 nm, 2 nm)
Spectra



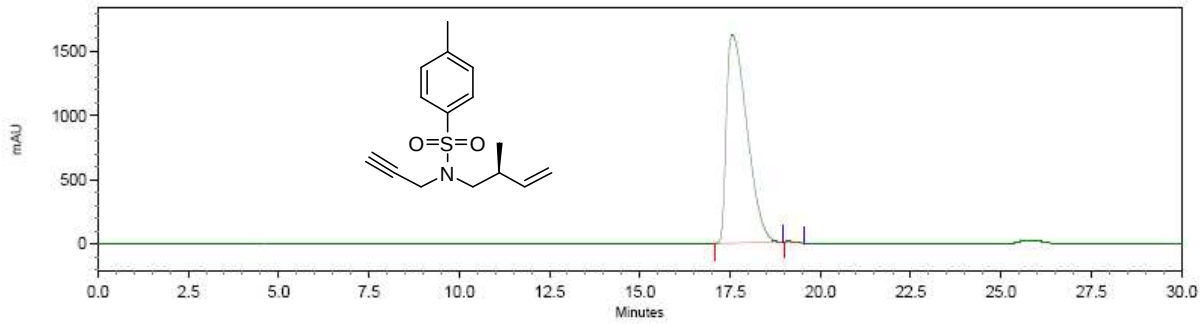
Retention time: 26,144 Min
Peak name: 1
Lambda max: 203, 231, 309
Lambda min: 342, 306, 218



Retention time: 27,584 Min
Peak name: 2
Lambda max: 203, 231, 309
Lambda min: 340, 302, 218

(S)-4-methyl-*N*-(2-methylbut-3-en-1-yl)-*N*-(prop-2-yn-1-yl)benzenesulfonamide (6a)

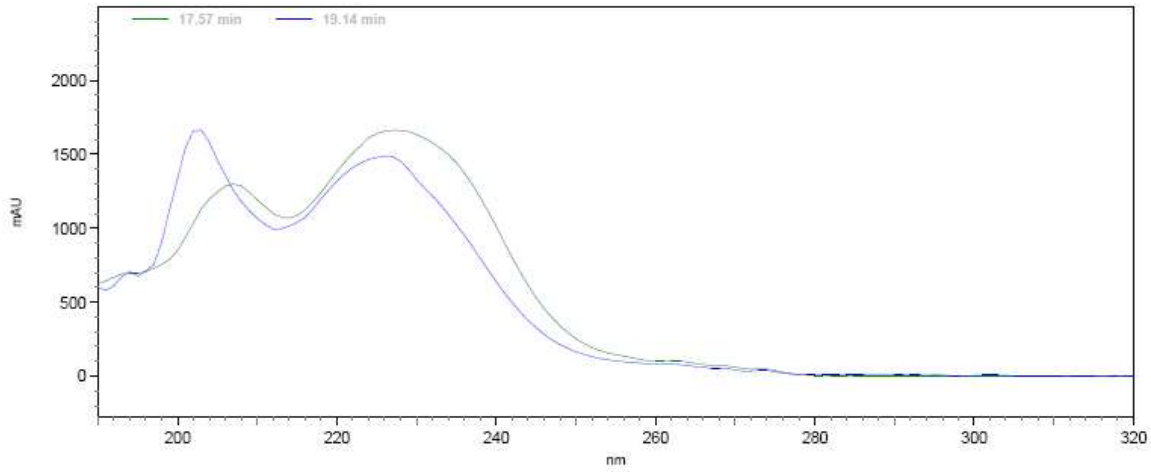
Sample ID:	**SZA016**	**Data Name:**	**F:\NMR\HPLC_data\20091211_02**
User:	**System**	**Method Name:**	**C:\CLASS-VP\Methods\Algemeen\Chiralpak AD\AD 99_1 30min.met**
Vial # :	**16**	**Inj. Vol :**	**1 ul**
Sample Amt:	**1**		
Acquired:	**12/11/2009 2:37:43 PM**	**Printed:**	**7/25/2010 6:11:34 PM**



1: 230 nm, 2 nm

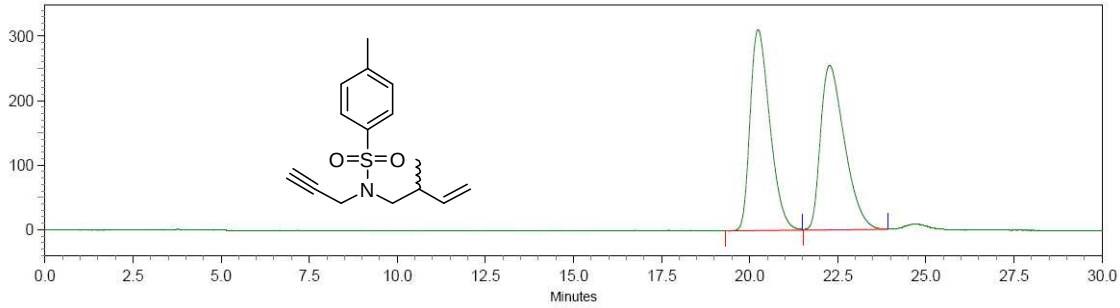
Pk #	Name	Retention Time	Area	Area Percent
1	1	17.568	64156986	99.87
2	2	19.136	85619	0.13
Totals			64242605	100.00

Overlaid Spectra



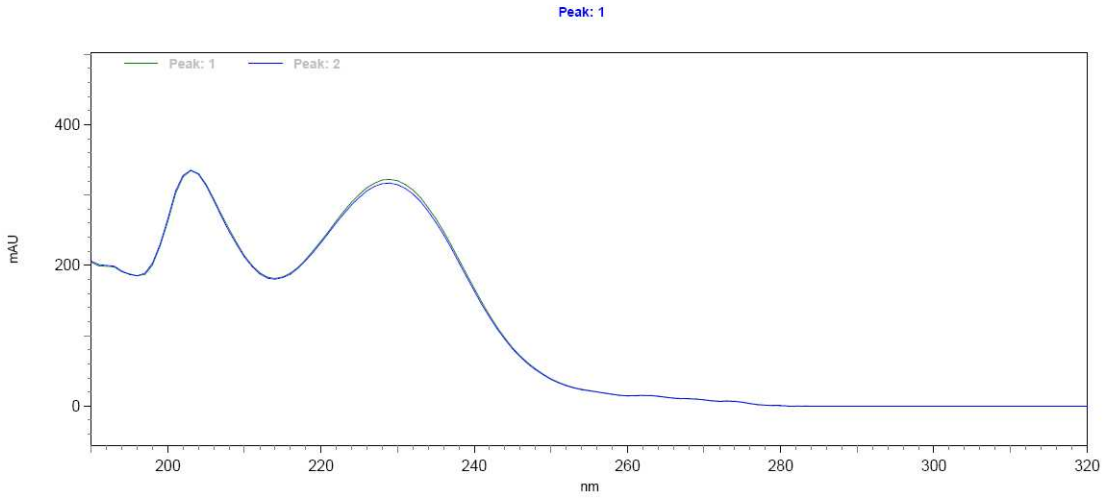
(**6a)** – Racemic

Sample ID: SZA017 **Data Name:** F:\NMR\20091210_15
User: System **Method Name:** C:\CLASS-VP\Methods\Algemeen\Chiralpak AD\AD 99_1 30min.met
Vial # : 17 **Inj. Vol :** 1 ul
Sample Amt: 1
Acquired: 12/10/2009 8:14:38 PM **Printed:** 7/16/2010 5:32:11 PM



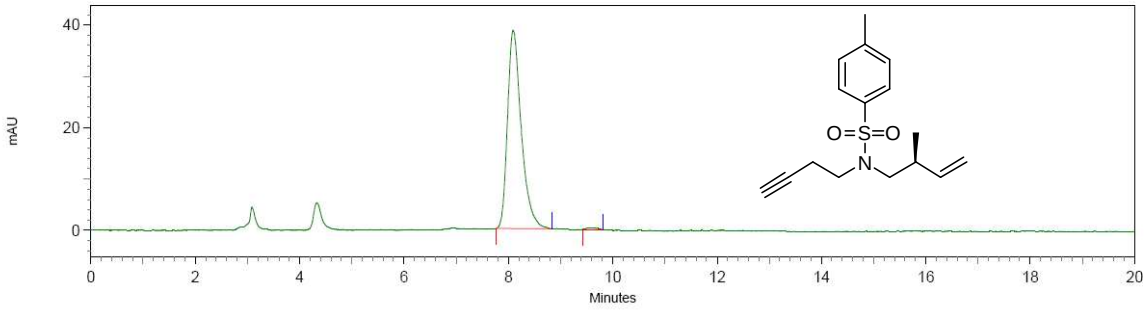
1: 230 nm, 8 nm

Pk #	Name	Retention Time	Area	Area Percent
1	1	20.235	12101228	50.00
2	2	22.272	12099511	50.00
Totals			24200739	100.00



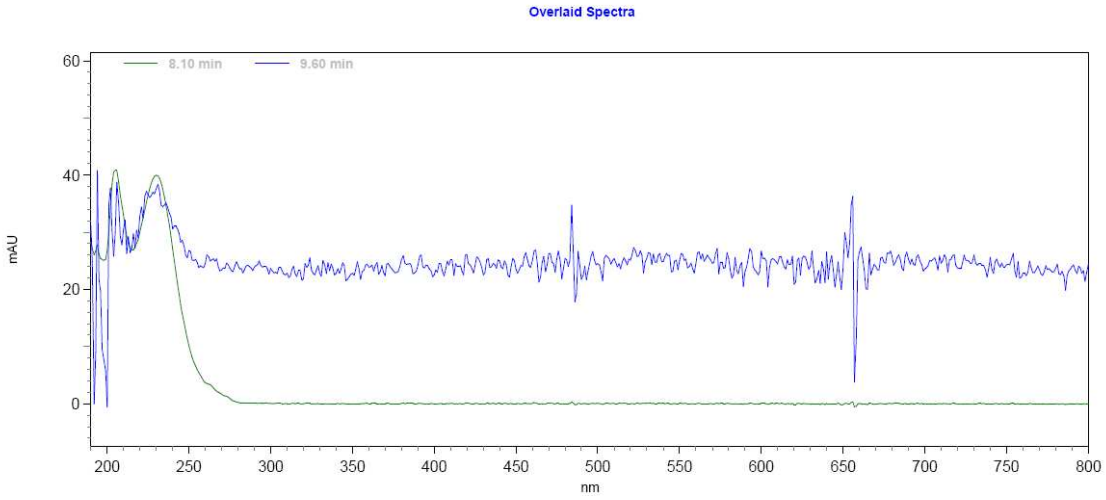
(S)-*N*-(but-3-yn-1-yl)-4-methyl-*N*-(2-methylbut-3-en-1-yl)benzenesulfonamide (6b)

Sample ID: SZA039 **Data Name:** C:\CLASS-VP\Data\20100219\20100219_05
User: System **Method Name:** C:\CLASS-VP\Methods\Algemeen\Chiralpak AD\AD 95_5 20min.met
Vial # : 23 **Inj. Vol :** 1 ul
Sample Amt: 1
Acquired: 2/19/2010 5:23:50 PM **Printed:** 7/16/2010 4:25:11 PM



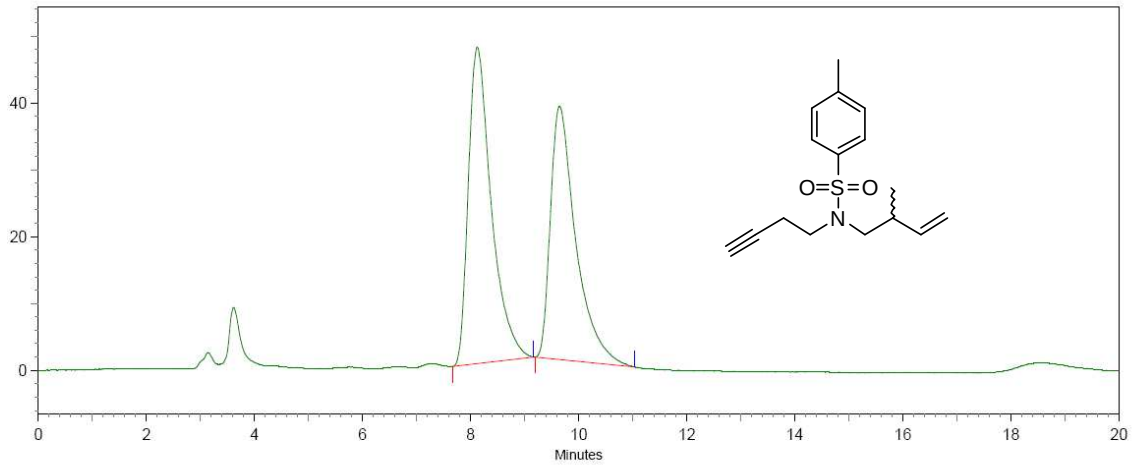
1: 230 nm, 8 nm

Pk #	Name	Retention Time	Area	Area Percent
1	1	8.096	705125	99.33
2	2	9.600	4750	0.67
Totals			709875	100.00



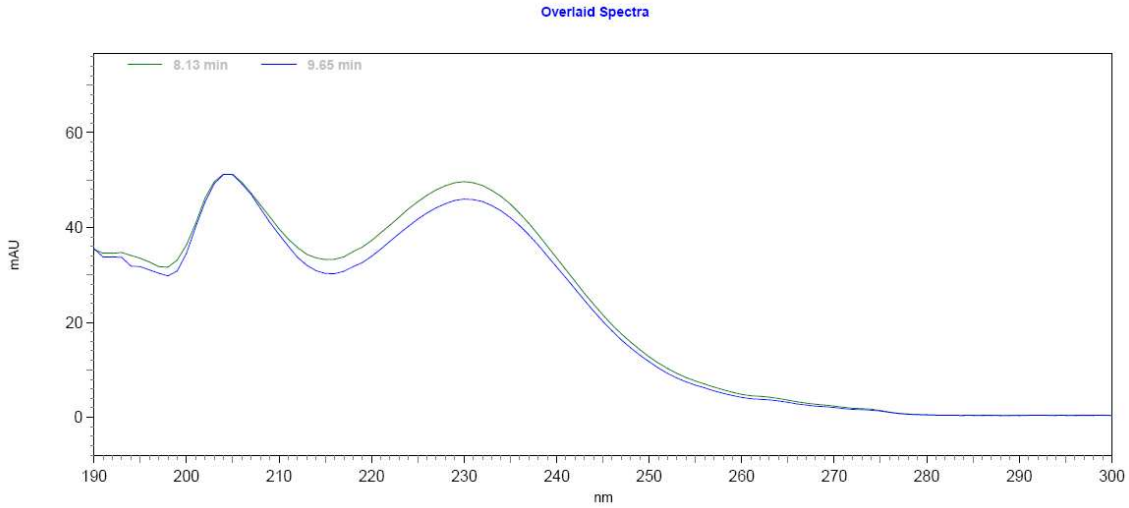
(6b) – Racemic

Sample ID:	**SZA041**	**Data Name:**	**C:\CLASS-VP\Data\20100324\20100323_04**
User:	**System**	**Method Name:**	**C:\CLASS-VP\Methods\Algemeen\Chiralpak AS\AS 95_5 20min.met**
Vial # :	**56**	**Inj. Vol :**	**1 ul**
Sample Amt:	**1**		
Acquired:	**3/24/2010 3:20:05 PM**	**Printed:**	**7/16/2010 4:49:43 PM**



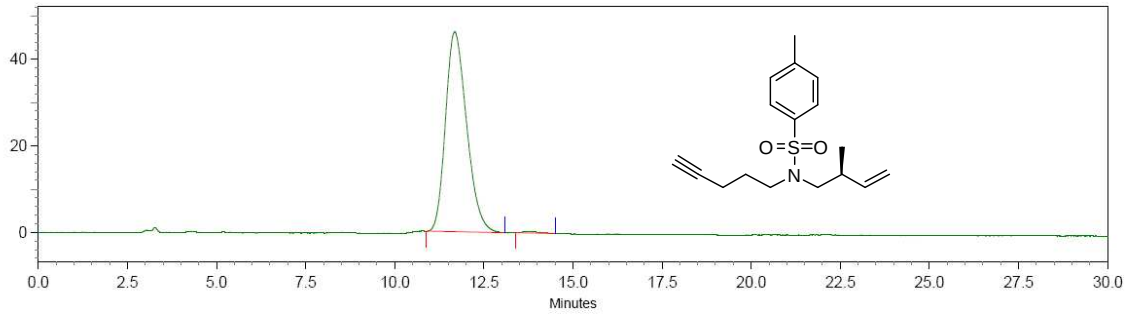
1: 230 nm, 8 nm

Pk #	Name	Retention Time	Area	Area Percent
1	1	8.128	1401594	53.20
2	2	9.653	1233223	46.80
Totals			2634817	100.00



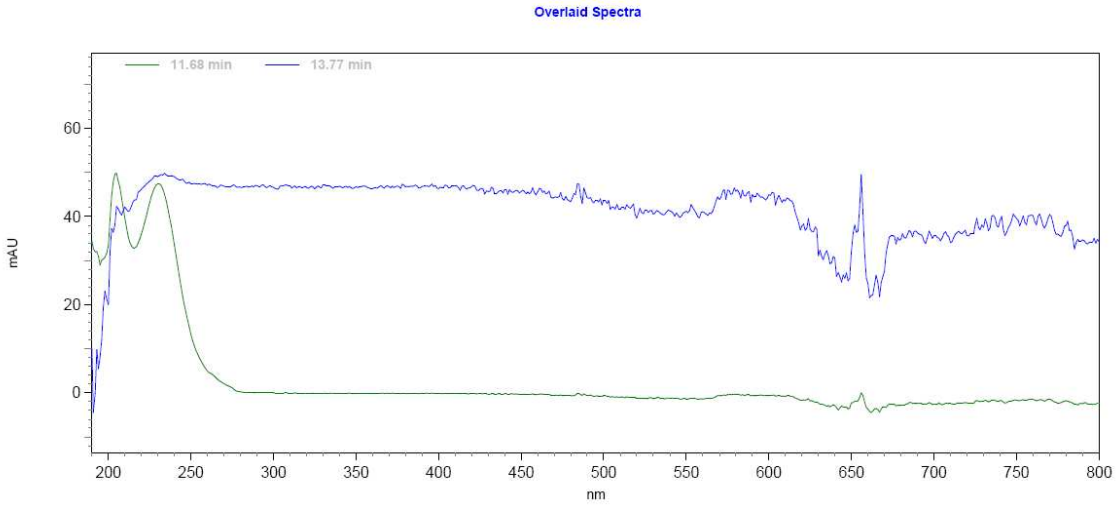
(S)-4-methyl-*N*-(2-methylbut-3-en-1-yl)-*N*-(pent-4-yn-1-yl)benzenesulfonamide (6c)

Sample ID:	**SZA092**	**Data Name:**	**C:\CLASS-VP\Data\20100714\20100714_05**
User:	**System**	**Method Name:**	**C:\CLASS-VP\Methods\Algemeen\Chiralcel OJ\OJ 97_3 30min.met**
Vial # :	**42**	**Inj. Vol :**	**1 ul**
Sample Amt:	**1**		
Acquired:	**7/14/2010 3:40:32 PM**	**Printed:**	**7/16/2010 4:54:13 PM**



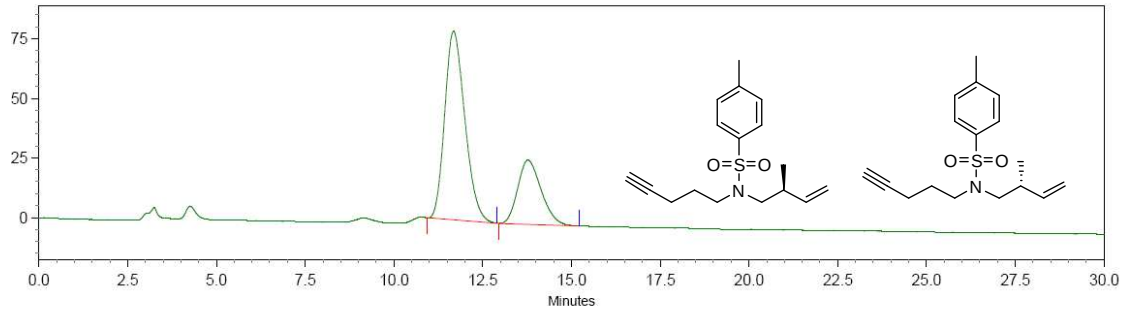
1: 230 nm, 8 nm

Pk #	Name	Retention Time	Area	Area Percent
1	1	11.680	1906401	99.42
2	2	13.728	11058	0.58
Totals			1917459	100.00



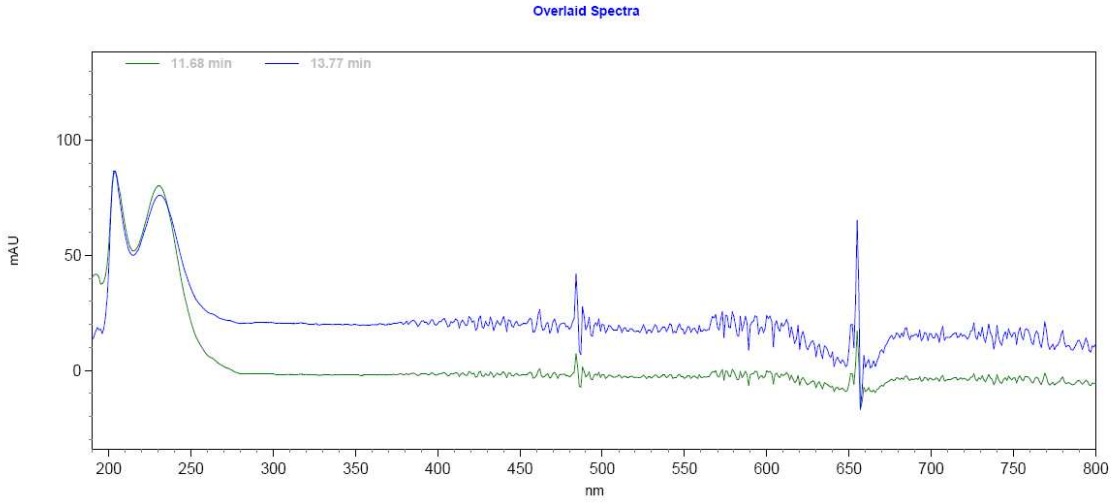
(6c) – Racemic

Sample ID:	**SZA073**	**Data Name:**	**C:\CLASS-VP\Data\20100714\20100714_03**
User:	**System**	**Method Name:**	**C:\CLASS-VP\Methods\Algemeen\Chiralcel OJ\OJ 97_3 30min.met**
Vial # :	**41**	**Inj. Vol :**	**1 ul**
Sample Amt:	**1**		
Acquired:	**7/14/2010 2:06:48 PM**	**Printed:**	**7/16/2010 4:53:31 PM**



1: 230 nm, 8 nm

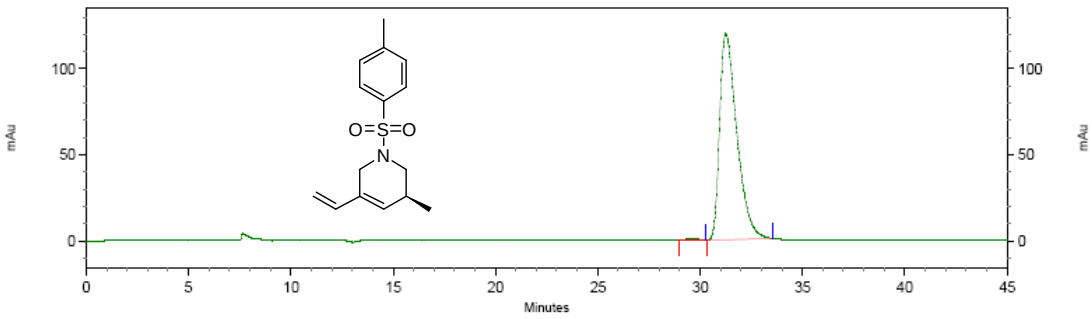
Pk #	Name	Retention Time	Area	Area Percent
1	1	11.680	3123015	71.62
2	2	13.771	1237746	28.38
Totals			4360761	100.00



(S)-3-Methyl-1-tosyl-5-vinyl-1,2,3,6-tetrahydropyridine (8a)

Sample ID : SZA019
Vial# : 19
Sample amount : 1
Inj. volume : 1
Acquired : 7-1-2010 9:39:58
Data Name : D:\DATA\20100106\20100106_02
Method Name :
C:\CLASS-VP\Enterprise\Projects\Default\Method\General\Chiralpak AS-H\AS-H 99_1 45 min.met
Sequence Name : C:\CLASS-VP\Enterprise\Projects\Default\Sequence\All 2009\20091130.seq

Chromatogram



1: 230
nm, 4 nm
Results

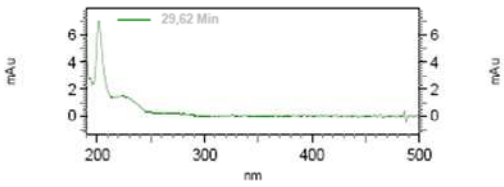
Pk #	Name	Retention Time	Area	Area Percent
1	Peak @ 29,624 Minutes	29,624	38385	0,544
2	Peak @ 31,256 Minutes	31,256	7017002	99,456
Totals			7055387	100,000

Spectrum Report

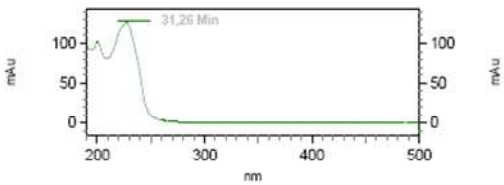
Spectra of all named detected peaks

(The peak spectrum is defined as the peak apex spectrum)

Multi-Chrom 1 (1: 230 nm, 4 nm)
Spectra



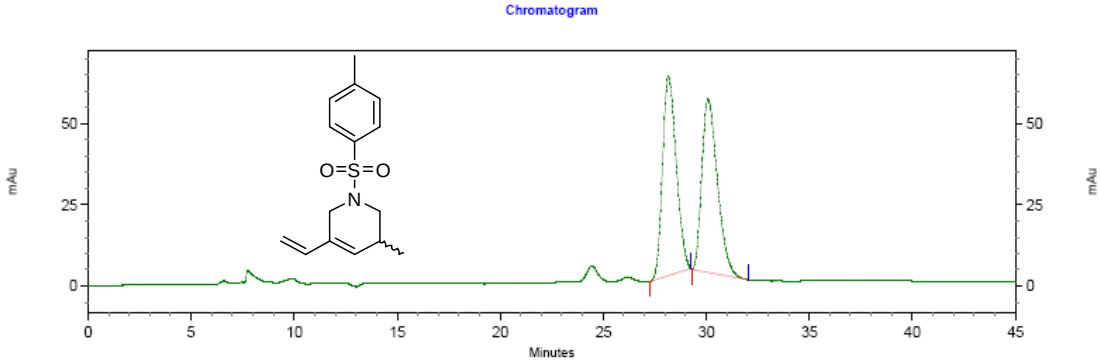
Retention time: 29,624 Min
Peak name: Peak @ 29,624 Minutes
Lambda max: 202, 223, 270
Lambda min: 494, 478, 435



Retention time: 31,256 Min
Peak name: Peak @ 31,256 Minutes
Lambda max: 227, 200, 320
Lambda min: 494, 460, 481

(8a) – Racemic

```
Sample ID      :    SZA018b
Vial#          :    18
Sample amount  :    1
Inj. volume    :    1
Acquired       :    7-1-2010 9:43:21
Data Name      :    D:\DATA\20100106\20100106_01
Method Name    :
C:\CLASS-VP\Enterprise\Projects\Default\Method\General\Chiralpak AS-H\AS-H
99_1 45 min.met
Sequence Name  :    C:\CLASS-VP\Enterprise\Projects\Default\Sequence\All
2009\20091130.seq
```



1: 230
nm, 4 nm
Results

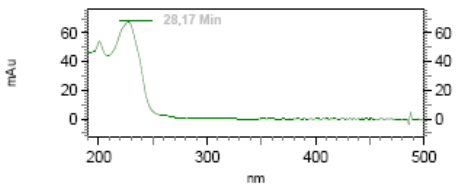
Pk #	Name	Retention Time	Area	Area Percent
1	Peak @ 28,172 Minutes	28,172	2855473	50,249
2	Peak @ 30,096 Minutes	30,096	2827128	49,751
Totals			5682601	100,000

Spectrum Report

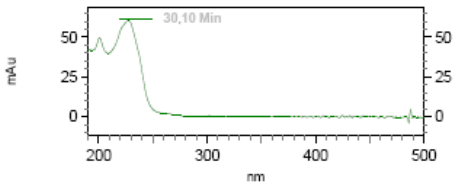
Spectra of all named detected peaks

(The peak spectrum is defined as the peak apex spectrum)

Multi-Chrom 1 (1: 230 nm, 4 nm)
Spectra



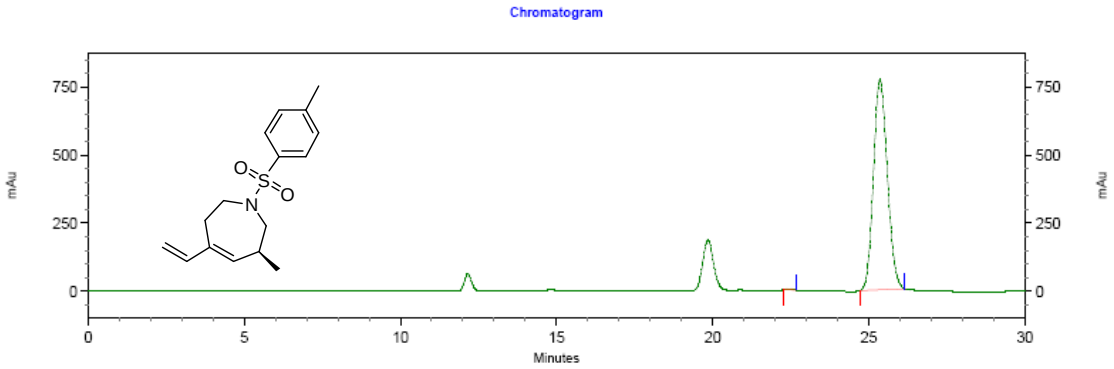
Retention time: 28,172 Min
Peak name: Peak @ 28,172 Minutes
Lambda max: 227, 201, 303
Lambda min: 434, 441, 405



Retention time: 30,096 Min
Peak name: Peak @ 30,096 Minutes
Lambda max: 227, 201, 303
Lambda min: 433, 441, 493

(S)-3-Methyl-1-tosyl-5-vinyl-2,3,6,7-tetrahydro-1H-azepine (8b)

Sample ID : SZA059
Vial# : 46
Sample amount : 1
Inj. volume : 1
Acquired : 6-5-2010 9:28:59
Data Name : D:\DATA\20100504\20100504_11
Method Name :
C:\CLASS-VP\Enterprise\Projects\Default\Method\General\Chiralpak OJ-H\OJ-H 95_5 30 min.met
Sequence Name :
C:\CLASS-VP\Enterprise\Projects\Default\Sequence\20100712.seq



1: 230
nm, 2 nm
Results

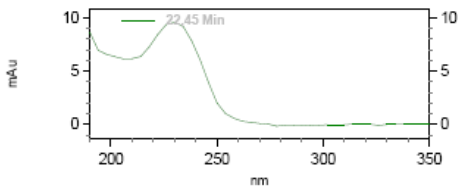
Pk #	Name	Retention Time	Area	Area Percent
1	1	22,452	64546	0,269
2	2	25,364	23903082	99,731
Totals			23967628	100,000

Spectrum Report

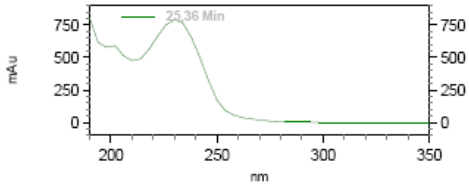
Spectra of all named detected peaks

(The peak spectrum is defined as the peak apex spectrum)

Multi-Chrom 1 (1: 230 nm, 4 nm)
Spectra



Retention time: 22,452 Min
Peak name: 1
Lambda max: 228
Lambda min: 289, 206

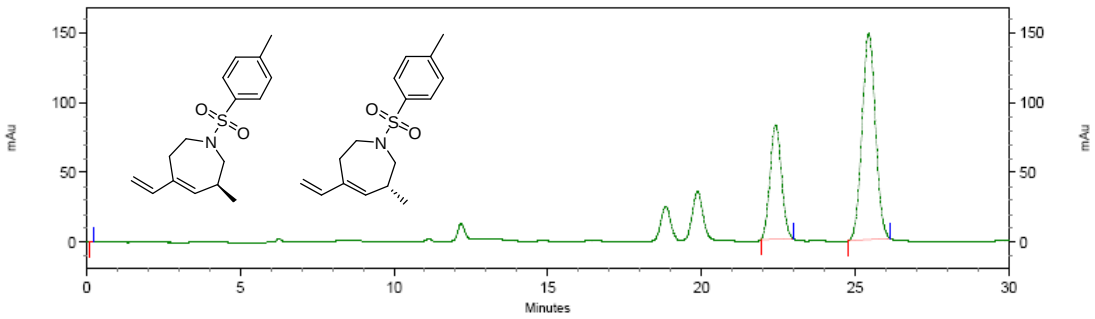


Retention time: 25,364 Min
Peak name: 2
Lambda max: 228
Lambda min: 208

(8b) – Racemic mixture

Sample ID : SZA059+69
Vial# : 50
Sample amount : 1
Inj. volume : 1
Acquired : 6-5-2010 9:24:04
Data Name : D:\DATA\20100504\20100504_15
Method Name :
C:\CLASS-VP\Enterprise\Projects\Default\Method\General\Chiralpak OJ-H\OJ-H 95_5 30 min.met
Sequence Name :
C:\CLASS-VP\Enterprise\Projects\Default\Sequence\20100712.seq

Chromatogram



1: 230
nm, 2 nm
Results

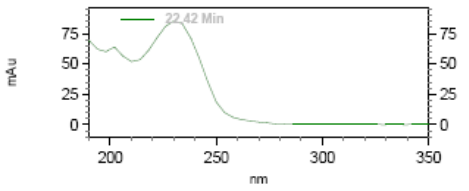
Pk #	Name	Retention Time	Area	Area Percent
2	1	22,424	2136964	32,281
3	2	25,448	4482720	67,717
Totals			6619684	99,998

Spectrum Report

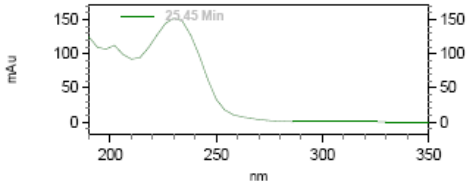
Spectra of all named detected peaks

(The peak spectrum is defined as the peak apex spectrum)

Multi-Chrom 1 (1: 230 nm, 4 nm)
Spectra



Retention time: 22,424 Min
Peak name: 1
Lambda max: 228
Lambda min: 343, 207



Retention time: 25,448 Min
Peak name: 2
Lambda max: 228
Lambda min: 343, 207